Supporting Information

Asymmetric Total Synthesis of Bioactive Natural Lipid Mycalol

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1. General Experimental Procedure: S2

2. NMR Comparison Tables: S(2-8)

3. HPLC Data for mixture of compound 15a and 15b: S9

4. Copies of Spectra: S(10-84)

General Experimental Procedure:

All moisture sensitive reactions were performed in oven or flame-dried glassware with Teflon coated magnetic stirring bar under argon atmosphere using dry, freshly distilled solvents, unless otherwise noted. Air- and moisture-sensitive liquids were transferred via a gastight syringe and a stainless-steel needle. Reactions were monitored by thin layer chromatography (TLC, Silica gel 60 F_{254}) plates with UV light, ethanolic anisaldehyde (with 1% AcOH and 3.3% conc. H_2SO_4)-heat and aqueous $KMnO_4$ (with K_2CO_3 and 10% aqueous NaOH solution) as developing agents. All workup and purification procedures were carried out with reagent-grade solvents under ambient atmosphere unless otherwise stated. Column chromatography was performed using silica gel 60-120 mesh and 100-200 mesh. Yields mentioned as chromatographically and spectroscopically homogeneous materials unless otherwise stated. Optical rotations were measured using sodium (589, D line) lamp and are reported as follows: $[\alpha]^{25}_{D}$ (*c* = mg/100 ml, solvent). IR spectra were recorded as thin films (for liquids). HRMS were taken using Quadruple-TOF (Q-TOF) micro MS system using electrospray ionisation (ESI) technique. ^{1}H NMR spectra were recorded on 300 and 500 MHz spectrometers in appropriate solvents and calibrated using residual undeuterated solvent as an internal reference, and the chemical shifts are shown in δ ppm scales. Multiplicities of NMR signals are designated as s (singlet), d (doublet), t (triplet), q (quartet), br s (broad singlet), m (multiplet, for unresolved lines) etc. ^{13}C spectra were recorded on 75, 100 and 125 MHz spectrometers.

Table 1: ^{13}C NMR comparison of mycalol (natural, C_5D_5N, 600 MHz), compound 1 (C_5D_5N, 300 MHz) δ in ppm.

Natural Mycalol	Compound 1
170.7	170.7
75.9	75.9
75.3,75.3	75.3
74.3	74.3
73.8	73.9
73.0	72.7
72.0	71.9
69.7	69.7
64.7	64.7
34.5,34.5	34.5,34.2
33.6,33.6	33.6
32.0	Merged with other signals
30.7	30.6
30.4	30.4
30.0,30.0,30.0,30.0	30.3,30.1,29.9,29.9,29.9,
29.8,29.8	29.8,27.8
26.7	26.8
25.7	25.7
25.3	Merged with other signals
22.8	22.8
21.1	21.1
14.1	14.1

Table 2: ^{13}C NMR comparison of triacetonide (natural, $CDCl_3$, 600 MHz), compounds 21, 22, 23, 24 ($CDCl_3$, 300 MHz) δ in ppm.

Natural triacetonide	Compound 21	Compound 22	Compound 23	Compound 24
171.0	171.1	171.1	171.1	171.1
109.4	109.6	109.6	109.6	109.5
107.6	107.8	107.8	107.8	107.8
107.4	107.5	107.6	107.6	107.5
78.3	78.5	78.2	78.2	78.5
78.2	78.4	74.9	77.6	78.4
78.1	78.2	74.8	74.9	78.2
74.8	74.9	74.8	74.8	74.9
74.6	74.9	74.6	74.6	74.6
74.5	74.6	Merged with other signals	Merged with other signals	Merged with other signals
71.9	72.3	72.3	72.3	72.1
68.7	68.8	68.9	68.9	68.8
66.9	67.0	66.9	66.9	67.0
34.1	34.3	34.3	34.3	34.3
34.0	33.9	33.9	33.9	33.9
31.7	Merged with other signals	32.1	Merged with other signals	Merged with other signals
30.2	30.3	30.2	30.2	30.3
29.7	29.8,29.7	29.8	29.8,29.8,29.7	29.8
29.5	29.1	29.8,29.7	29.5	29.7,29.7
28.6	28.7	28.7	28.7	28.8,28.7
27.2	27.6,27.2	27.6	28.7	27.6
26.8	26.9	26.9	27.8	27.3
26.4	26.5	26.6,26.5	26.6	26.9
26.0	26.1	26.2	26.2	26.5
25.9	26.1	26.1	26.1,26.1	26.1
25.4	25.5	25.6	25.6	25.6
25.3	25.5	25.5	25.5	25.5
25.0	Merged with other signals	Merged with other signals	Merged with other signals	Merged with other signals
22.5	22.7	22.8	22.8	22.8
21.3	21.4	21.4	21.4	21.4
14.1	14.1	14.1	14.1	14.1

Table 3: ^{13}C NMR comparison of triacetonide (natural, $CDCl_3$, 600 MHz), compounds 43, 44, 45, 46 ($CDCl_3$, 300 MHz) δ in ppm.

Natural triacetonide	Compound 43	Compound 44	Compound 45	Compound 46
171.0	171.1	171.1	171.1	171.1
109.4	109.5	109.5	109.5	109.6
107.6	107.8	107.8	107.8	107.7
107.4	107.6	107.6	107.5	107.5
78.3	78.5	78.2	78.5	78.5
78.2	78.3	Merged with other signals	78.4	78.4
78.1	78.2	Merged with other signals	78.2	78.2
74.8	74.8	75.0	75.0	74.9
74.6	74.6	74.8	74.8	74.9
74.5	74.5	74.6	74.6	Merged with other signals
71.9	72.1	72.1	72.1	72.6
68.7	68.8	68.9	68.9	68.8
66.9	67.0	66.9	67.0	67.0
34.1	34.3	34.3	34.3	34.3
34.0	33.9	33.9	33.9	33.9
31.7	Merged with other signals	32.1	Merged with other signals	Merged with other signals
30.2	30.3	30.2	30.3	30.3
29.7	29.8,29.7,29.7	29.9,29.8,29.7	29.8	29.8,29.8,29.8
29.5	29.5	29.5	29.8	29.7,29.5
28.6	28.7	28.7	28.7	28.8
27.2	27.6	27.6	27.6	27.3
26.8	26.9	26.9	27.3	26.9
26.4	26.5	26.6	27.2	26.5
26.0	26.1	26.4		26.1
25.9	26.0	26.1	26.1	26.1
25.4	25.6	25.6	25.5	25.6
25.3	25.5	25.5	25.5	25.5
25.0	Merged with other signals	Merged with other signals	Merged with other signals	Merged with other signals
22.5	22.8	22.8	22.8	22.7
21.3	21.4	21.4	21.4	21.4
14.1	14.1	14.1	14.1	14.1

Table 4: ^{13}C NMR comparison of triacetonide (natural, $CDCl_3$, 600 MHz), compounds 53, 54 ($CDCl_3$, 300 MHz) δ in ppm.

Natural triacetonide	Compound 53	Compound 54
171.0	171.1	171.1
109.4	109.6	109.6
107.6	107.7	107.7
107.4	107.5	107.4
78.3	78.2	78.2
78.2	77.8	78.1
78.1	77.8	77.9
74.8	74.9	74.9
74.6	74.8	74.8
74.5	74.6	74.6
71.9	72.2	72.6
68.7	68.9	68.8
66.9	66.9	66.9
34.1	34.3	34.3
34.0	33.9	33.9
31.7	32.1	Merged with other signals
30.2	30.3	30.3
29.7	29.8	29.8
29.5	29.7,29.5	29.7,29.7
28.6	28.7	28.8
27.2	27.6	27.6
26.8	26.9	26.9
26.4	26.4	26.6
26.0	26.2	26.6
25.9	26.1	26.1
25.4	25.6	25.6
25.3	25.5	25.5
25.0	Merged with other signals	Merged with other signals
22.5	22.8	22.8
21.3	21.5	21.4
14.1	14.1	14.1

Table 5: ^{13}C NMR comparison of triacetonide (natural, $CDCl_3$, 600 MHz), compounds 65, 66 ($CDCl_3$, 300 MHz) δ in ppm.

Natural triacetonide	Compound 65	Compound 66
171.0	171.1	171.1
109.4	109.6	109.5
107.6	107.8	107.8
107.4	107.5	107.5
78.3	78.5	78.5
78.2	78.3	78.4
78.1	78.2	78.2
74.8	74.9	74.9
74.6	74.8	74.8
74.5	74.6	74.6
71.9	72.3	72.1
68.7	68.8	68.8
66.9	67.0	67.0
34.1	34.3	34.3
34.0	Merged with other signals	34.2
31.7	31.9	31.9
30.2	30.3	30.3
29.7	29.8,29.7	29.8
29.5	29.3	29.7
28.6	28.7	28.7
27.2	27.3	27.2
26.8	26.9	26.9
26.4	26.5	26.5
26.0	26.1	26.1
25.9	25.9	25.9
25.4	25.5	25.6
25.3	25.4	25.5
25.0	Merged with other signals	25.1
22.5	22.7	22.7
21.3	21.4	21.4
14.1	14.2	14.1

Table 6: ^{1}H and ^{13}C NMR comparison of mycalol (natural, C_5D_5N, 600 MHz), compound 2 (C_5D_5N, 300 MHz) δ in ppm.

^{1}H NMR of Natural Mycalol	^{1}H NMR of Compound 2	^{13}C NMR of Natural Mycalol	^{13}C NMR of Compound 2
5.07, m, 1H	5.13-5.05, m, 1H (Merged with water signal)	170.7	170.7
4.35, m, 1H	4.38-4.34, m, 1H	75.9	75.9
4.18, m, 1H	4.24-4.18, m, 1H	75.3,75.3	75.2
4.08, m, 2H 4.06, m, 1H 4.05, m, 1H	4.14-4.07, m, 4H	74.3	74.2
3.99, m, 1H 3.97, m, 2H	4.04-3.94, m, 3H	73.8	73.7
3.90, *dd*, 9.5, 5.0 Hz, 1H 3.85, *dd*, 9.5, 6.0 Hz, 1H	3.93-3.82, m, 2H	73.0	72.9
2.59, *bd*, 8.2 Hz, 2H	2.57, *bd*, 9.3 Hz, 2H	72.0	71.9
2.40, m, 1H	2.41-2.35, m, 1H	69.7	69.6
2.16, m, 1H 2.11, m, 2H 2.09, s, 3H	2.18-2.09, m, 6H	64.7	64.7
1.99, m, 1H 1.87, m, 1H 1.86, m, 1H	1.98-1.79, m, 3H	34.5,34.5	34.5,34.4
1.56, m, 5H	1.58-1.50, m, 5H	33.6,33.6	33.6
1.40-1.26, m, 8H 1.36, m, 2H 1.33, m, 4H 1.22, m, 4H 1.20, m, 2H	1.34-1.21, m, 20H	32.0	31.9
0.82, *t*, 6.7 Hz, 3H	0.81, *t*, 6.9 Hz, 3H	30.7	30.6
		30.4	30.3
		30.0,30.0,30.0,30.0	30.0,29.8
		29.8,29.8	29.8,29.8
		26.7	26.7
		25.7	25.7
		25.3	25.3
		22.8	22.7
		21.1	21.1
		14.1	14.1

HPLC Data for mixture of compound 15a and 15b:

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\sds-mc-admix1.lcd

Acquired by	: Admin
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Sample ID	: sds-mc-admix
Vail #	: 1
Injection Volume	: 15 uL
Data File Name	: sds-mc-admix1.lcd
Method File Name	: HEX-IPA 95-5 40 min.lcm
Batch File Name	: SingleRun120141014223818.lcb
Report File Name	: Default.lcr
Data Acquired	: 10/14/2014 10:39:01 PM
Data Processed	: 10/22/2014 10:04:25 AM

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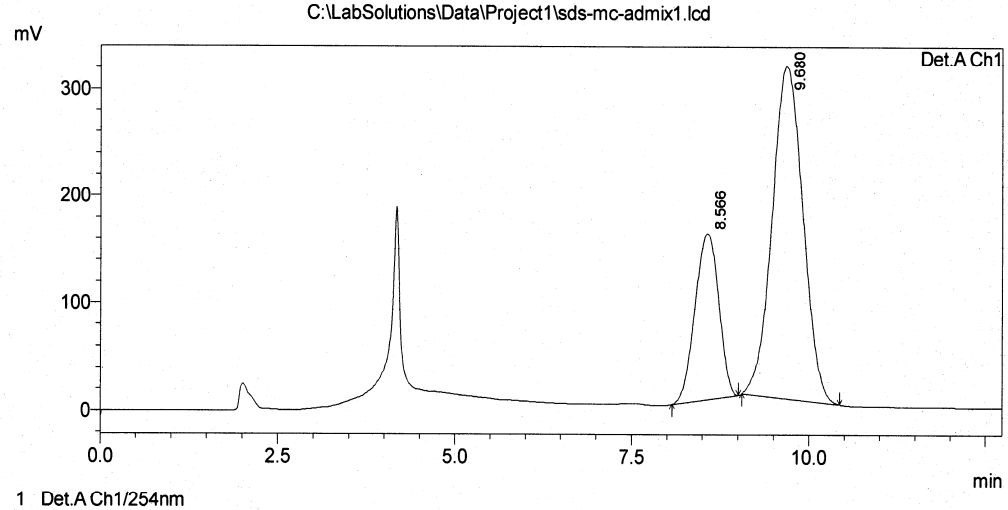


1 Det.A Ch1/254nm

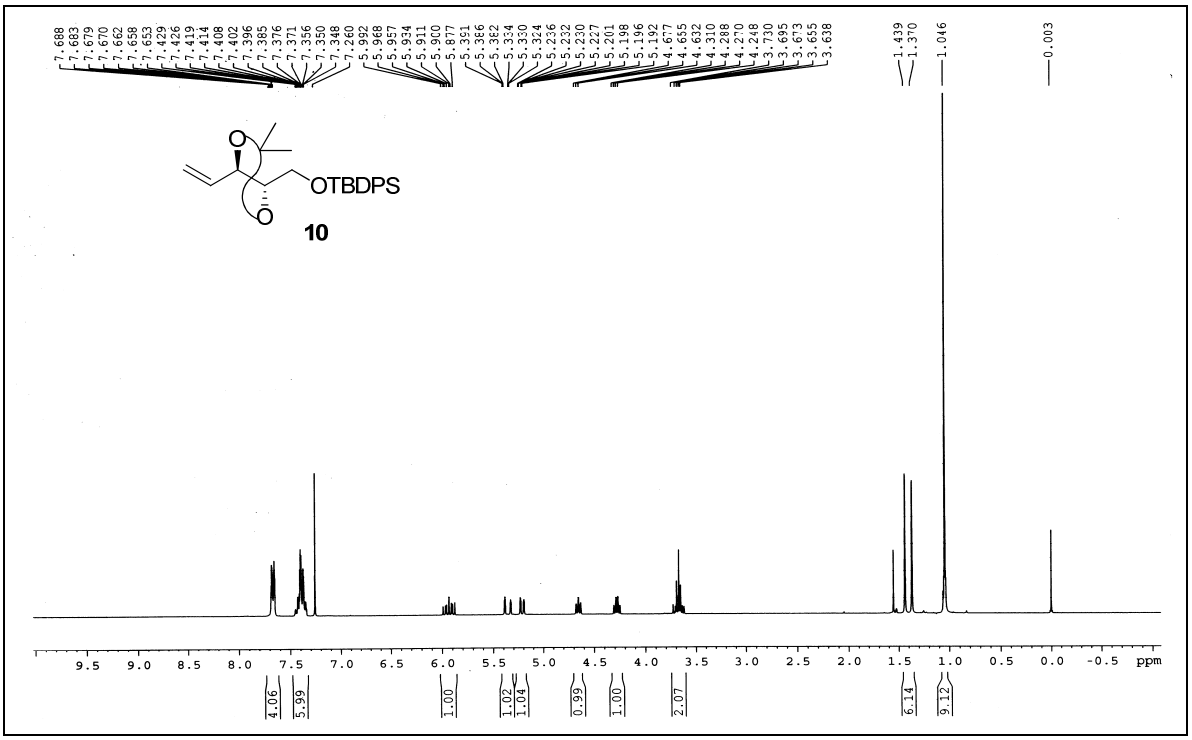
PeakTable

Detector A Ch1 254nm

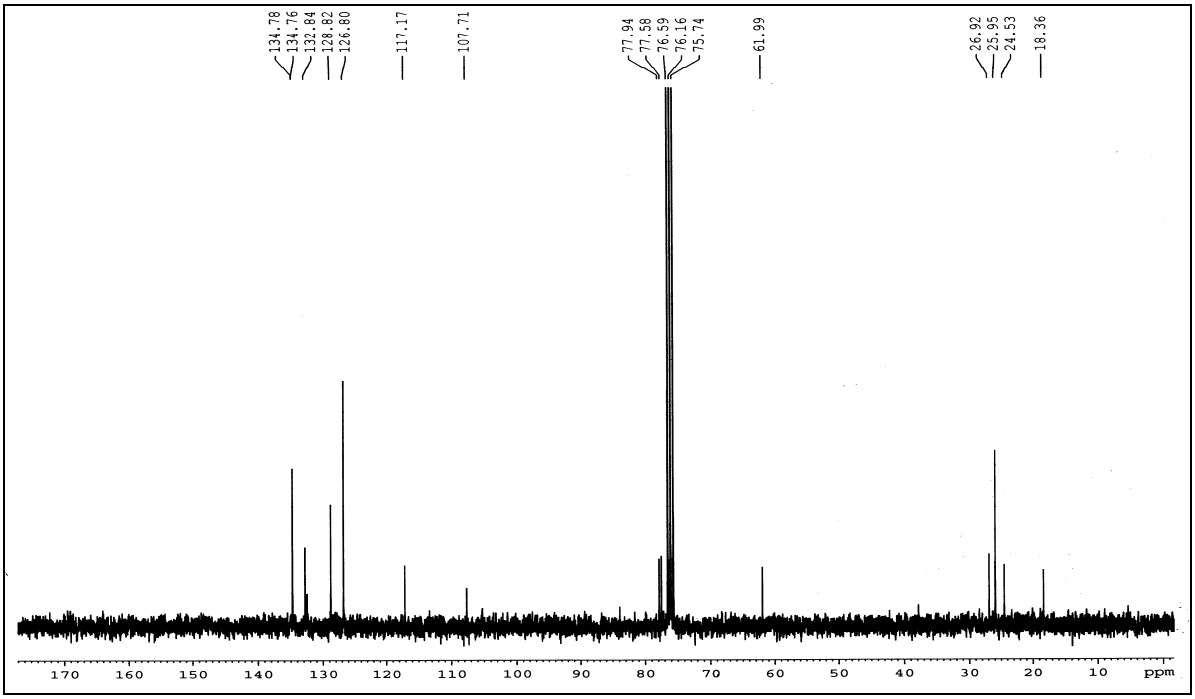
Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.566	3585291	154645	27.750	33.282
2	9.680	9334643	310008	72.250	66.718
Total		12919934	464653	100.000	100.000

Spectra (^{1}H NMR, ^{13}C NMR, HRMS) of compounds:

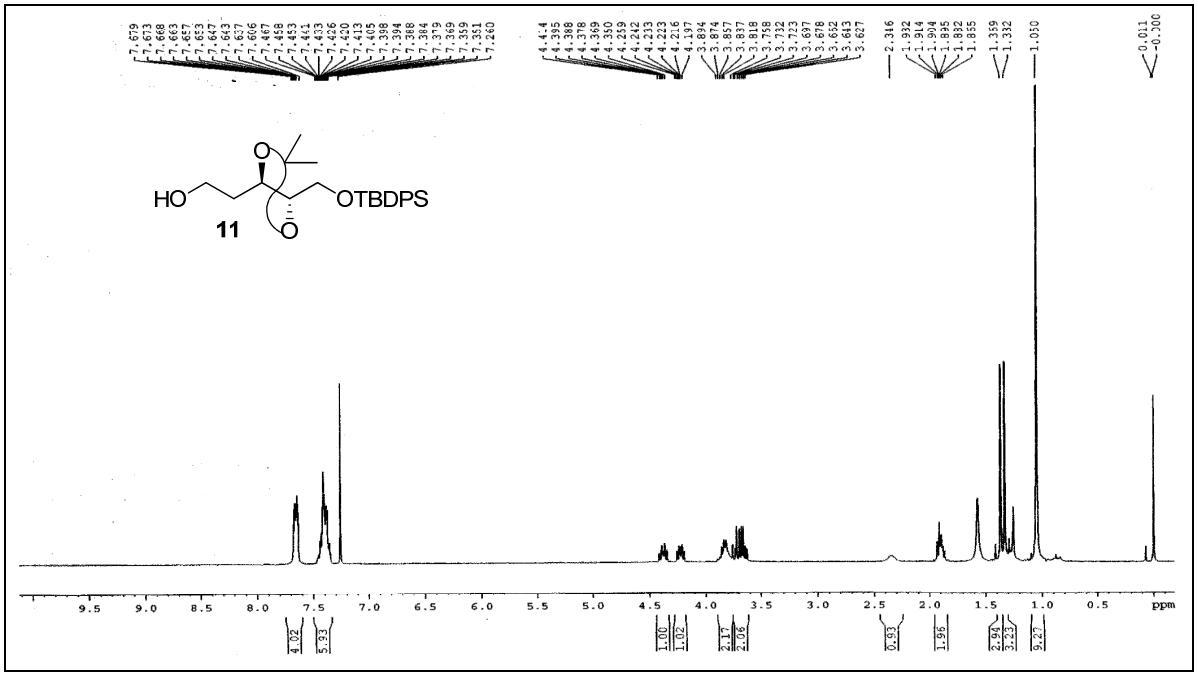
^{1}H NMR spectrum of 10 (300 MHz, $CDCl_3$):



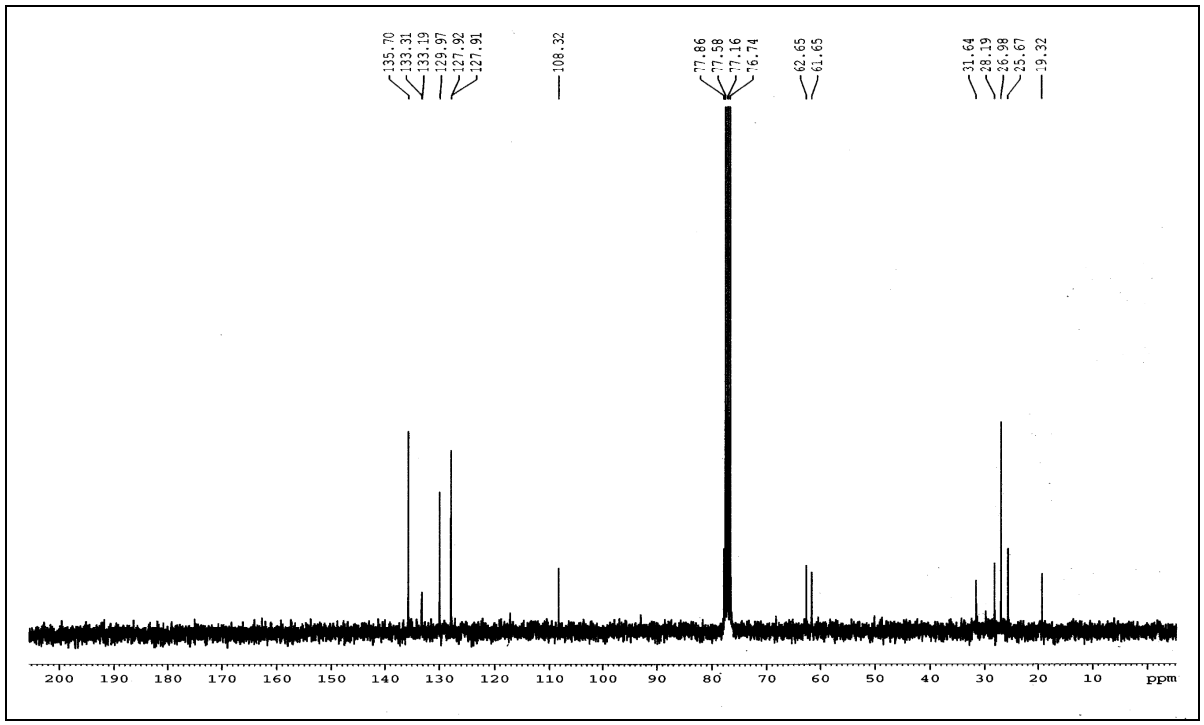
^{13}C NMR spectrum of 10 (75 MHz, $CDCl_3$):



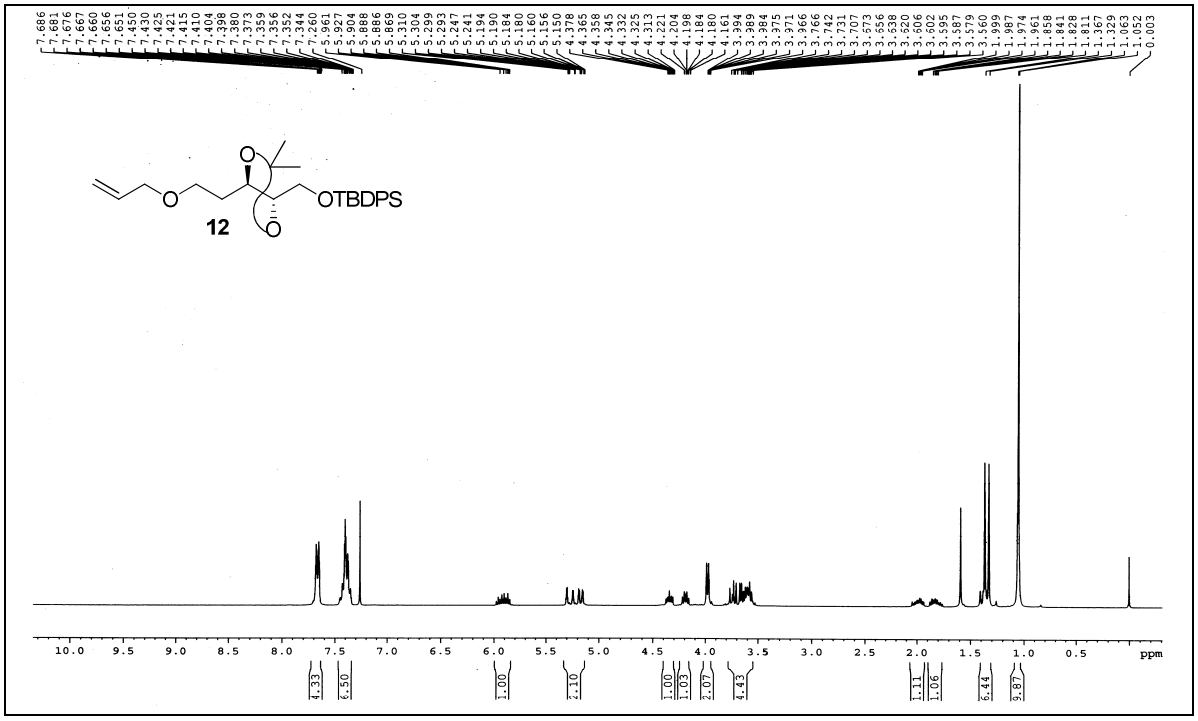
^{1}H NMR spectrum of 11 (300 MHz, $CDCl_3$):



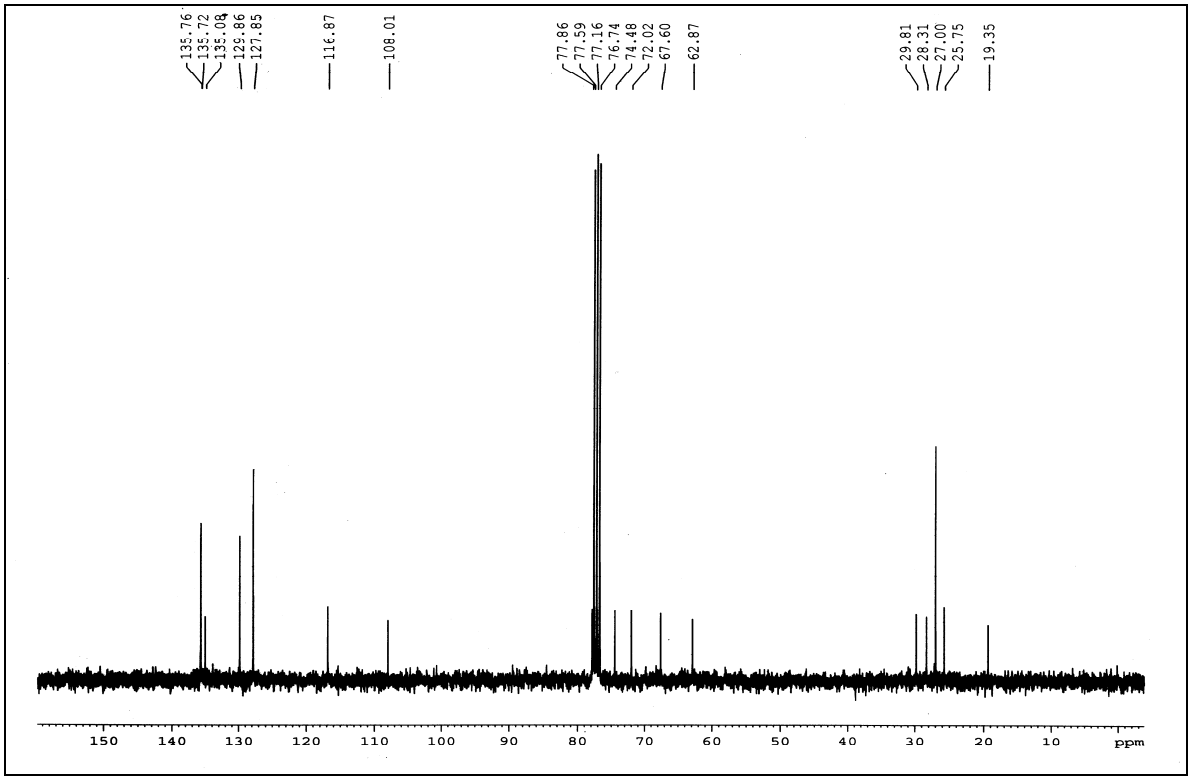
^{13}C NMR spectrum of 11 (75 MHz, $CDCl_3$):



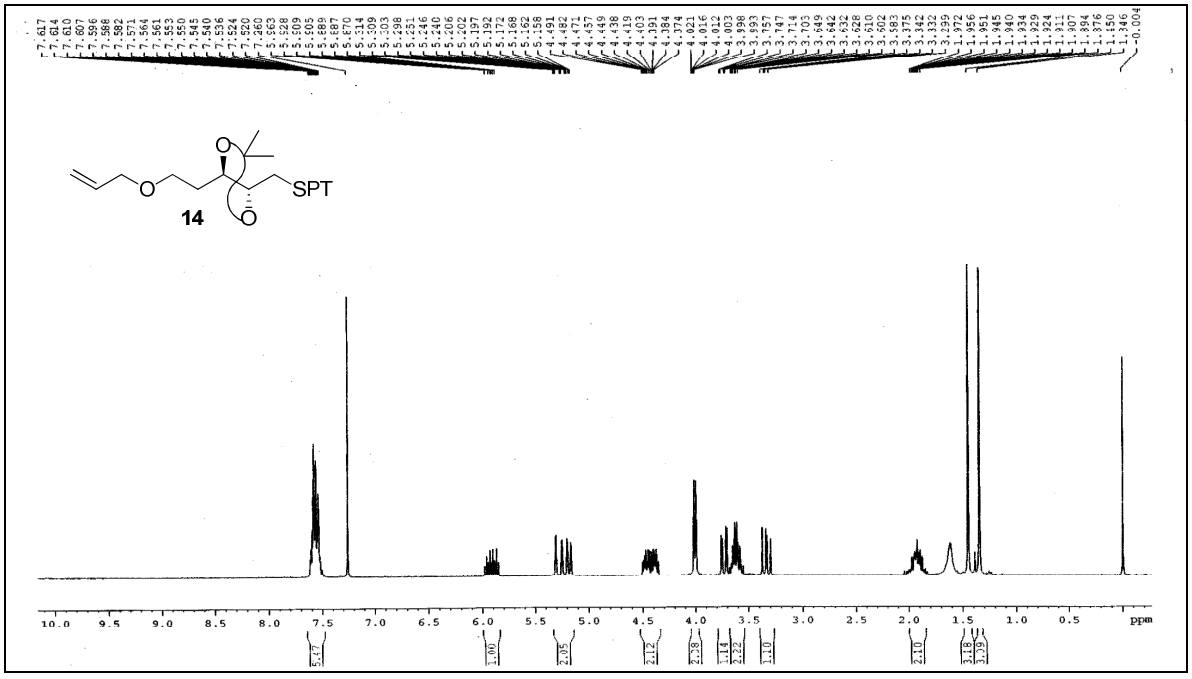
^{1}H NMR spectrum of 12 (300 MHz, $CDCl_3$):



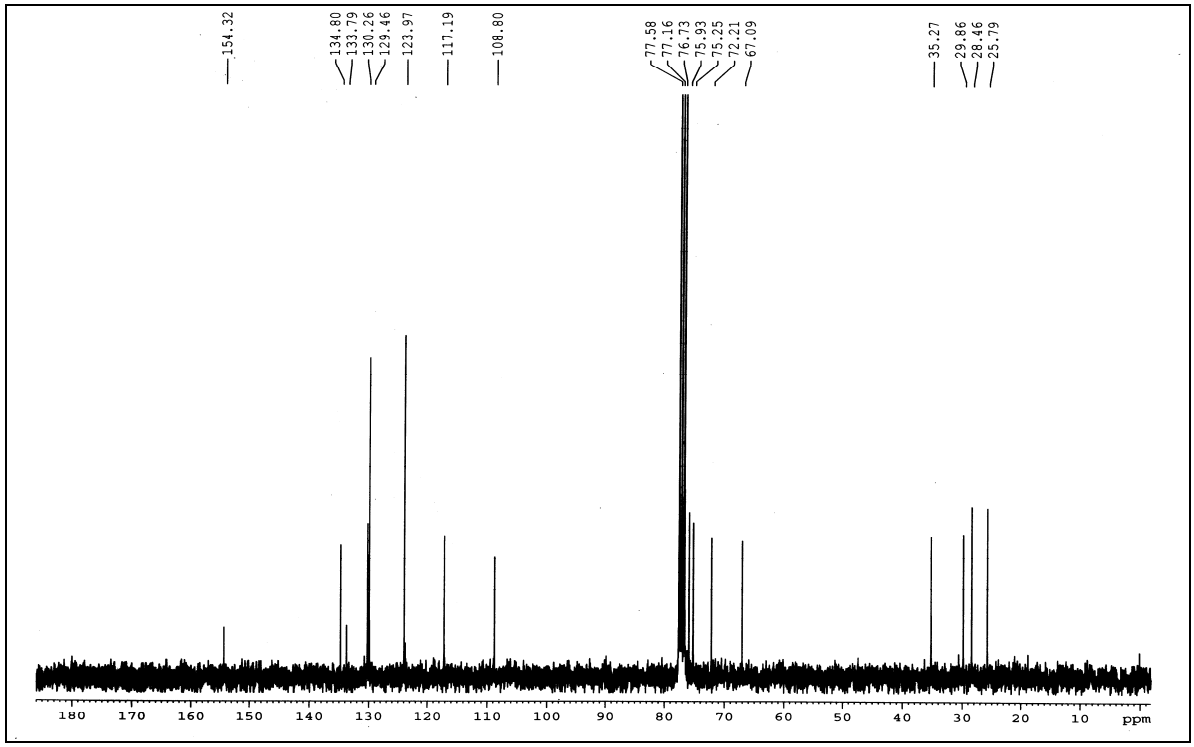
^{13}C NMR spectrum of 12 (75 MHz, $CDCl_3$):



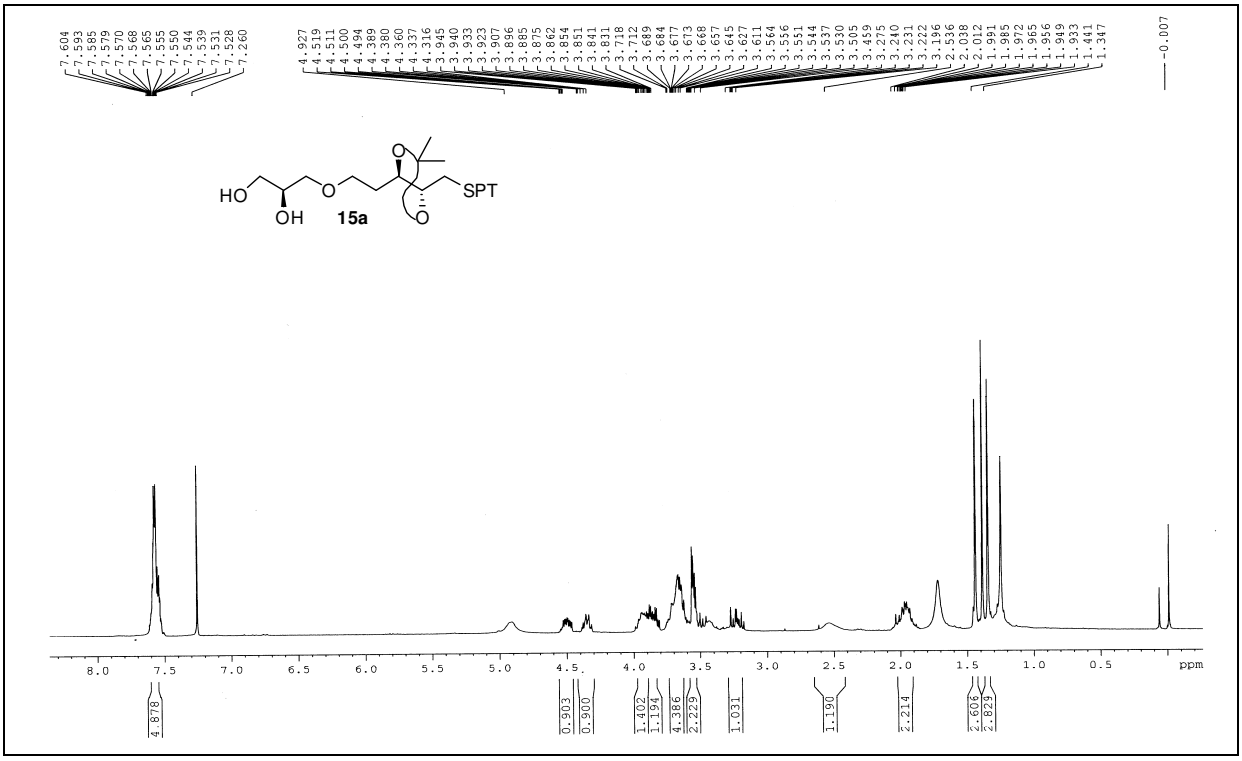
^{1}H NMR spectrum of 14 (300 MHz, $CDCl_3$):



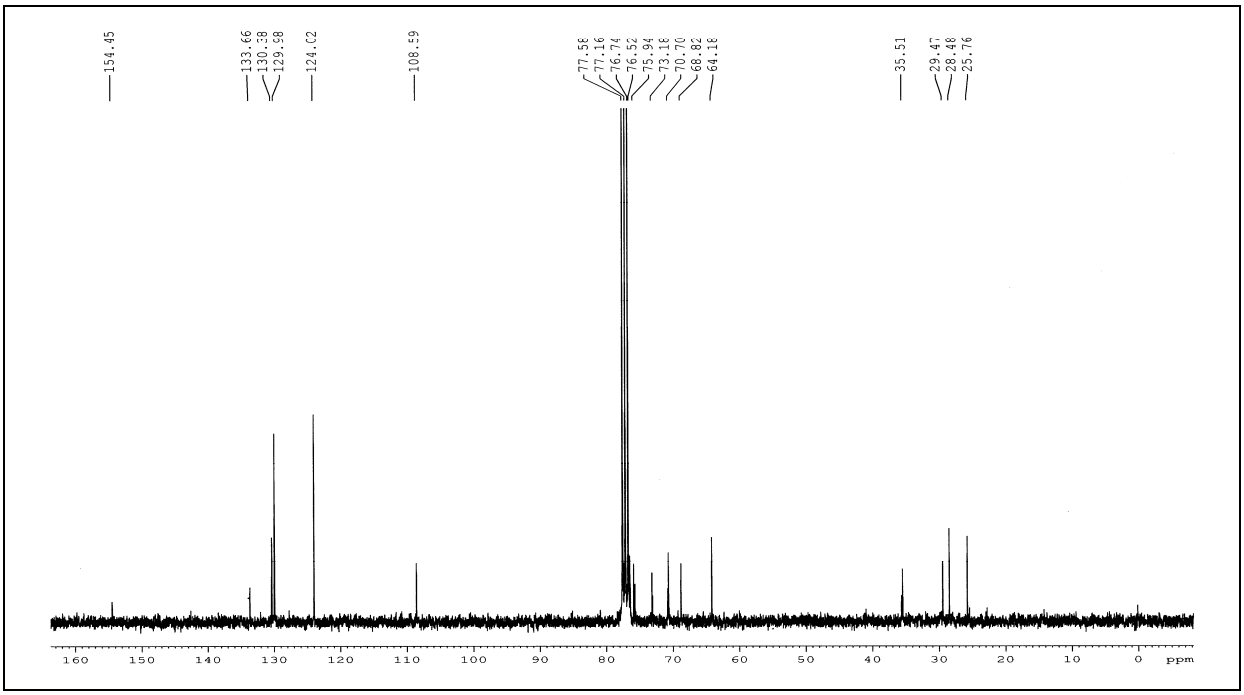
^{13}C NMR spectrum of 14 (75 MHz, $CDCl_3$):



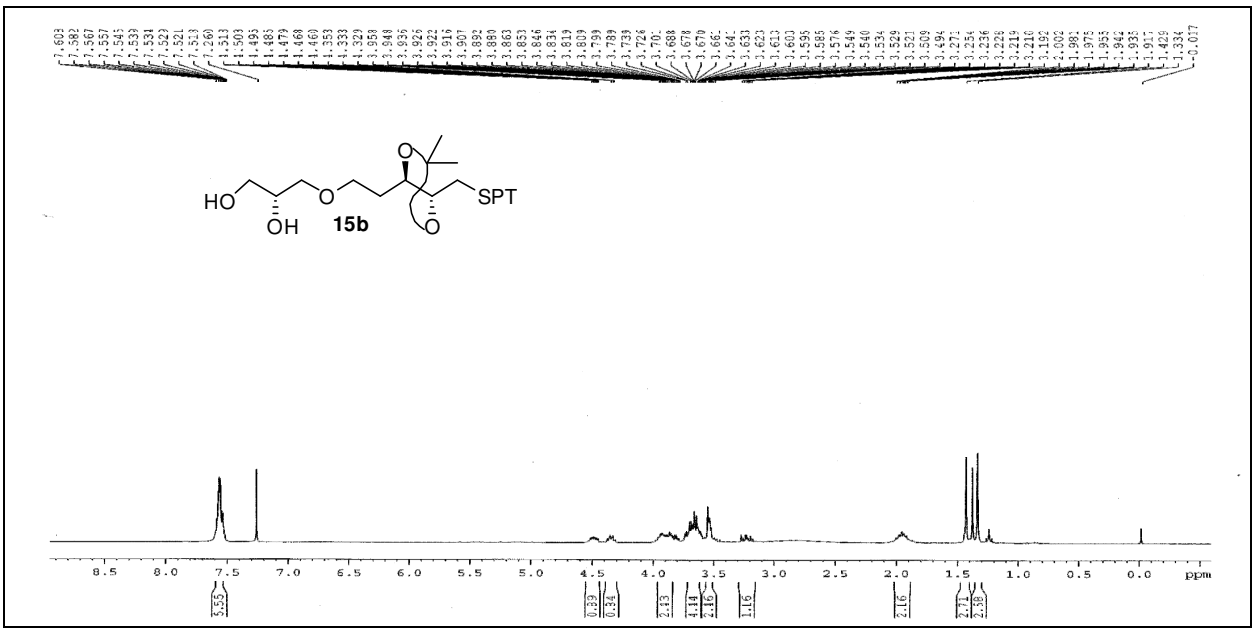
^{1}H NMR spectrum of 15a (300 MHz, $CDCl_3$):



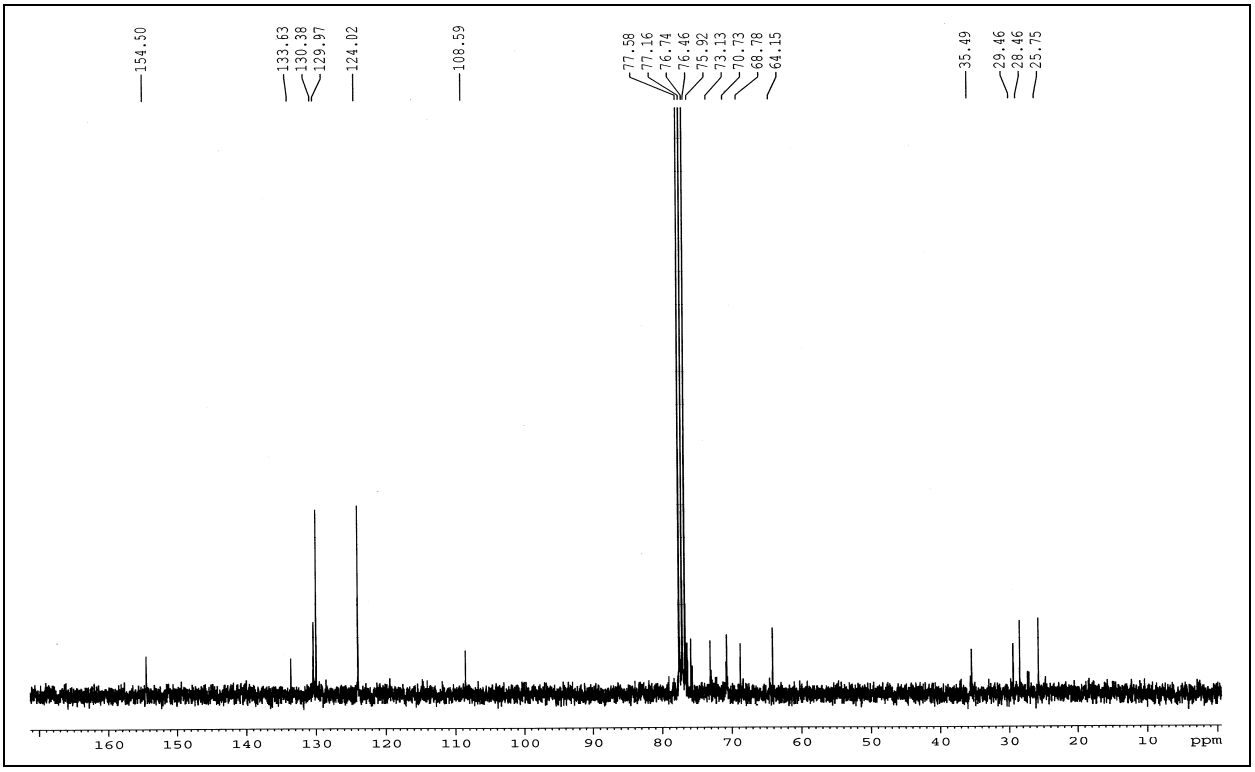
^{13}C NMR spectrum of 15a (75 MHz, $CDCl_3$):



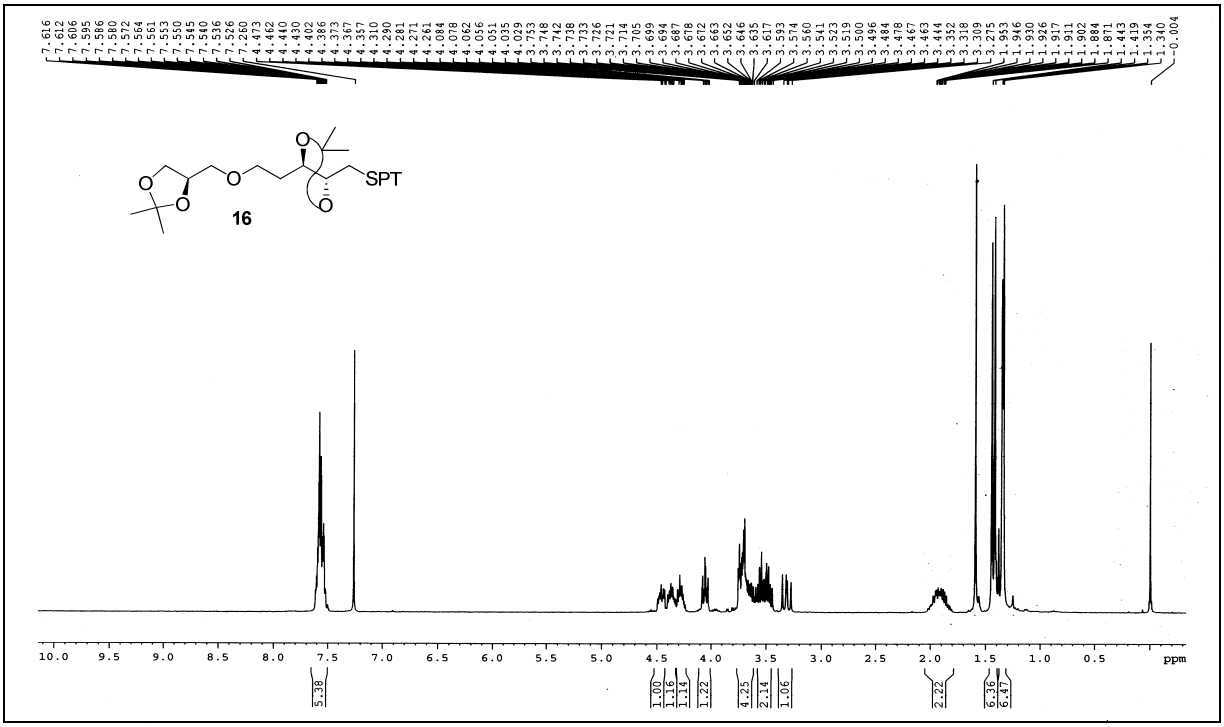
^{1}H NMR spectrum of 15b (300 MHz, $CDCl_3$):



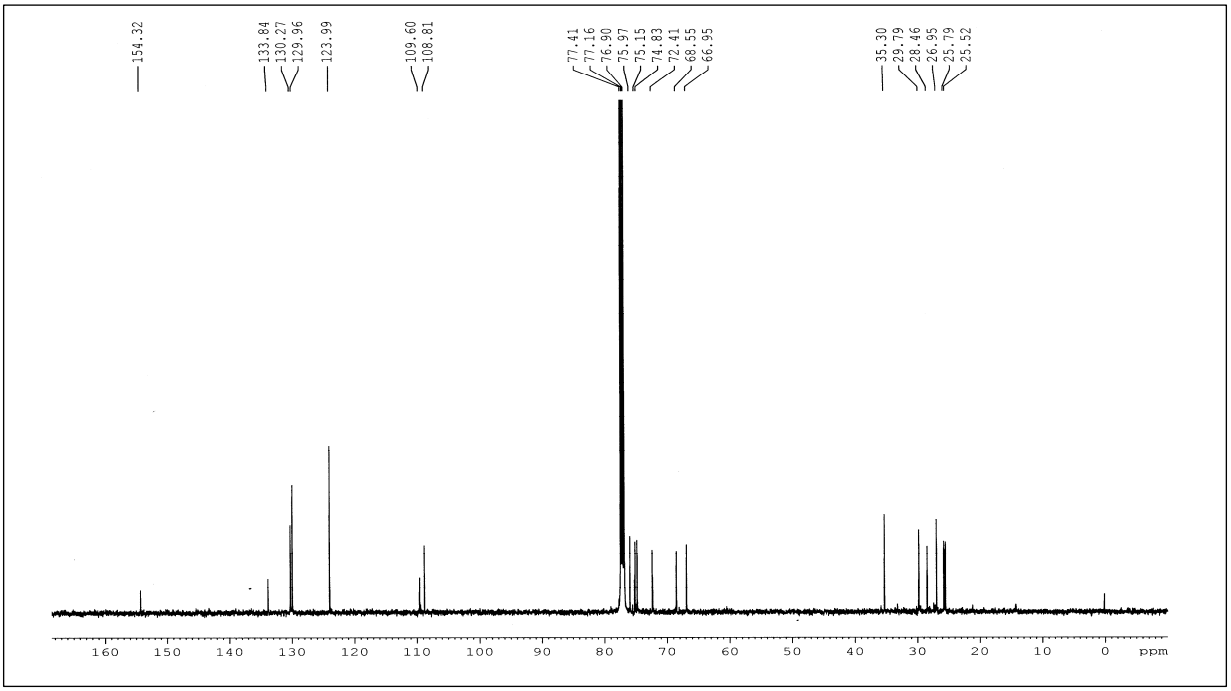
^{13}C NMR spectrum of 15b (125 MHz, $CDCl_3$):



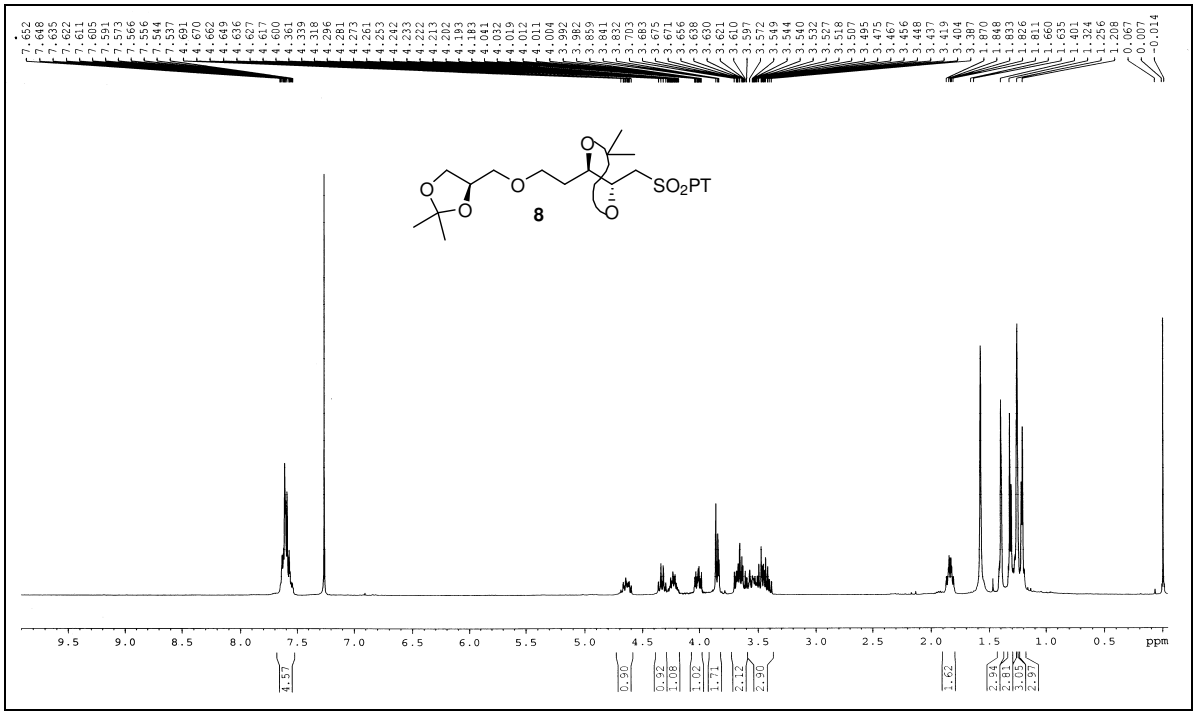
^{1}H NMR spectrum of 16 (300 MHz, $CDCl_3$):



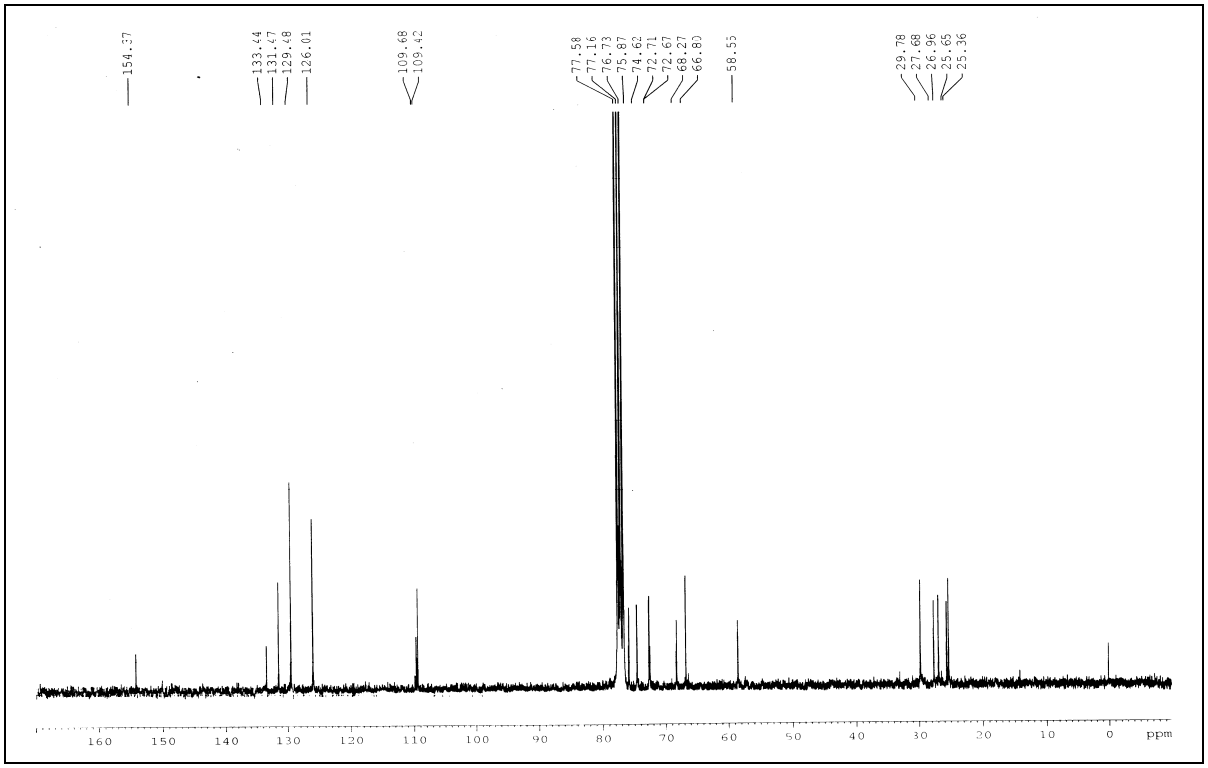
^{13}C NMR spectrum of 16 (125 MHz, $CDCl_3$):



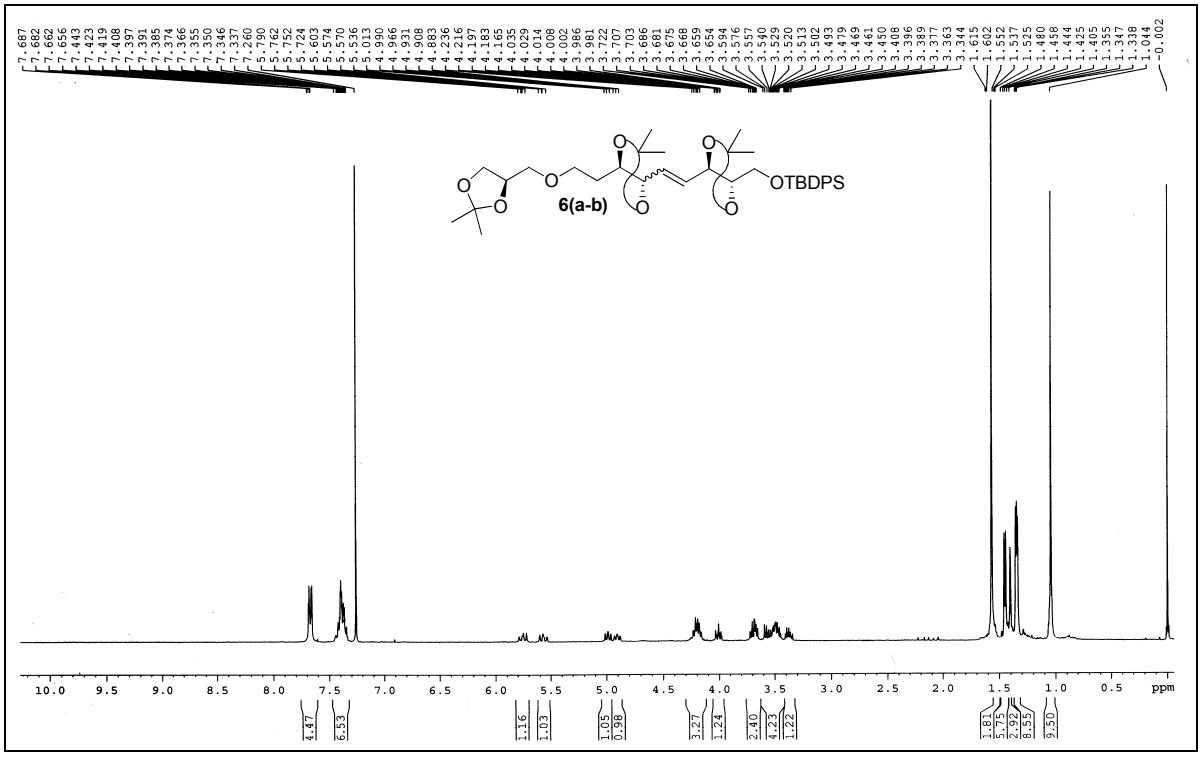
^{1}H NMR spectrum of 8 (300 MHz, $CDCl_3$):



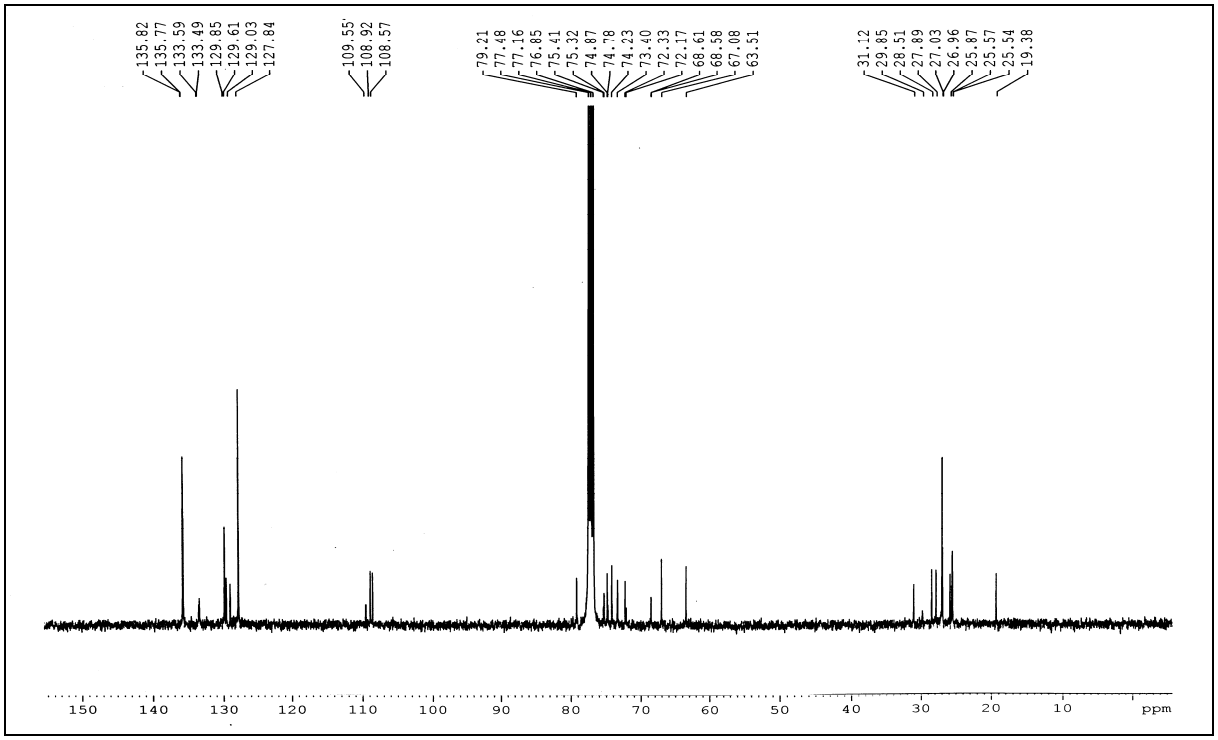
^{13}C NMR spectrum of 8 (75 MHz, $CDCl_3$):



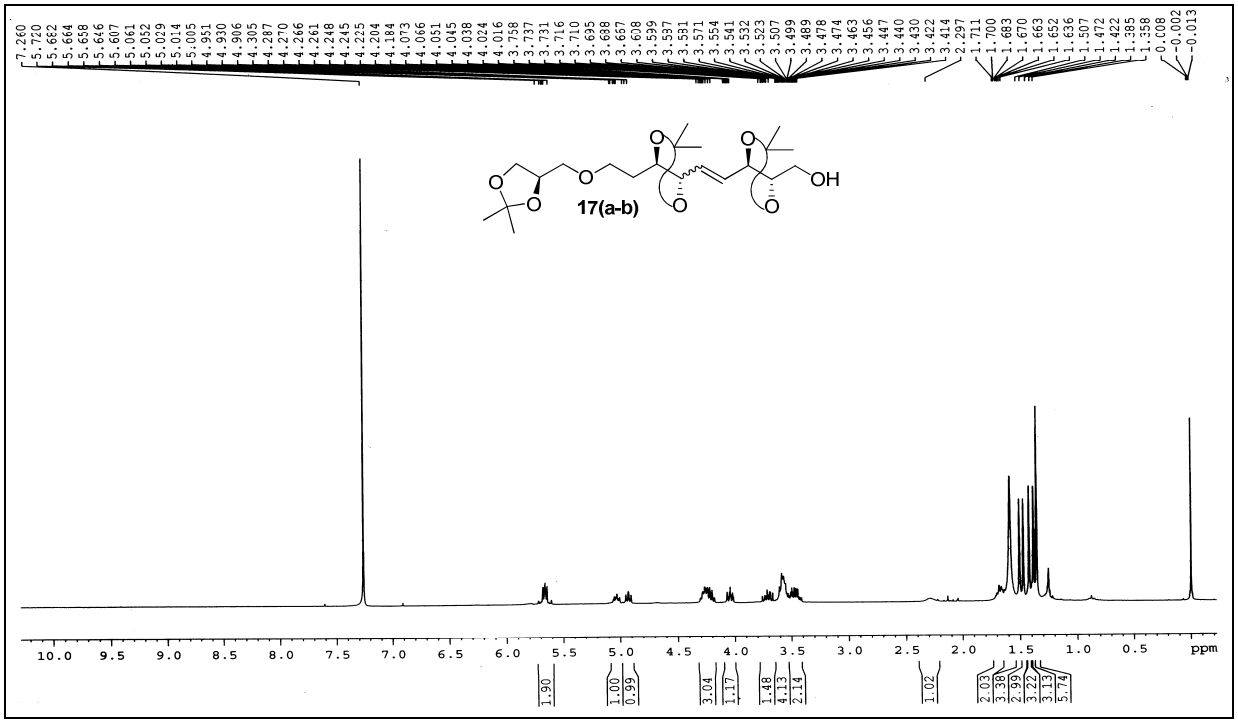
^{1}H NMR spectrum of diastereomaric mixture of 6(a-b) (300 MHz, $CDCl_3$):



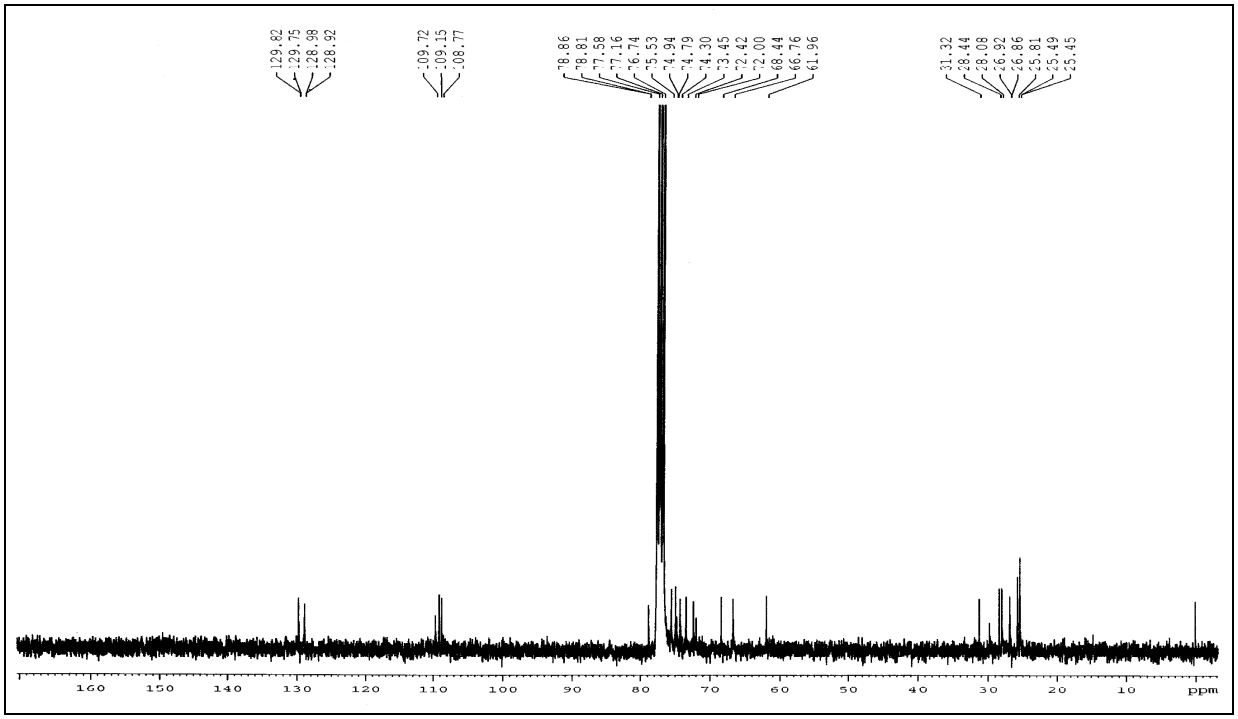
^{13}C NMR spectrum of diastereomaric mixture of 6(a-b) (75 MHz, $CDCl_3$):



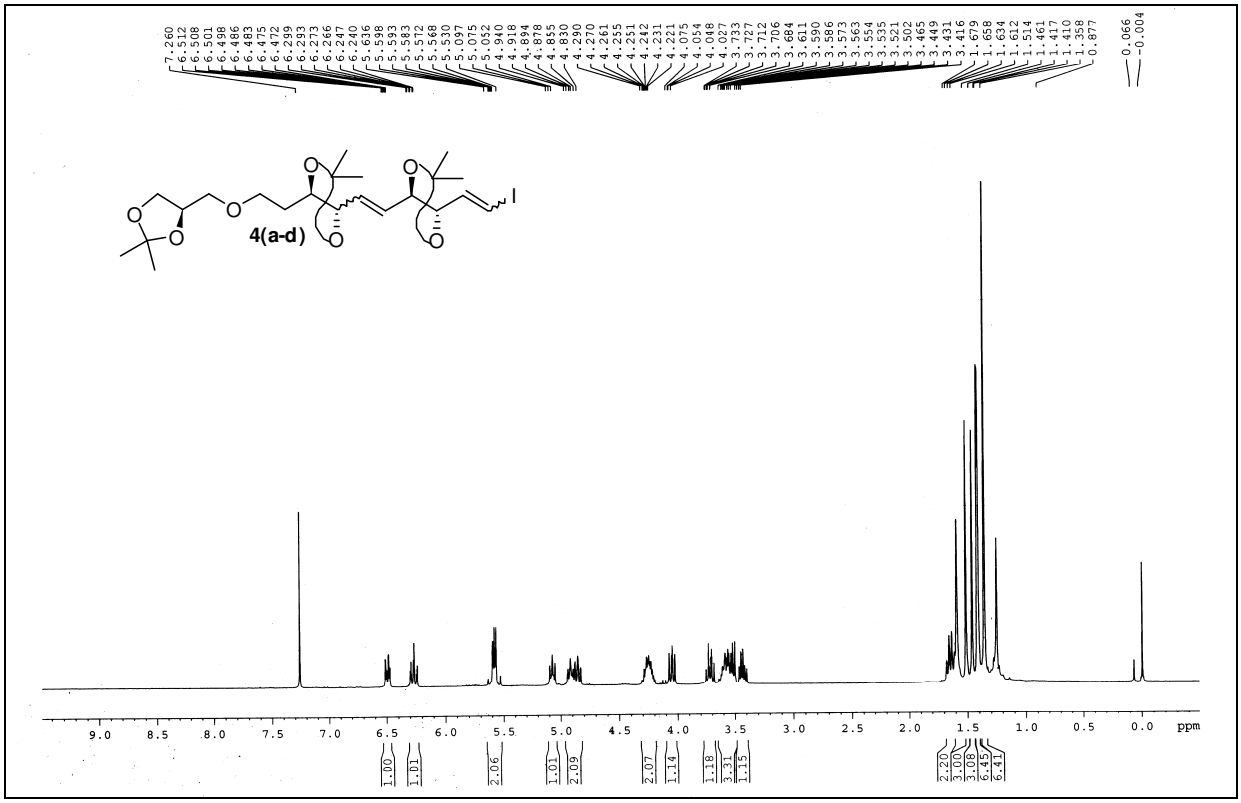
^{1}H NMR spectrum of diastereomaric mixture of 17(a-b) (300 MHz, $CDCl_3$):



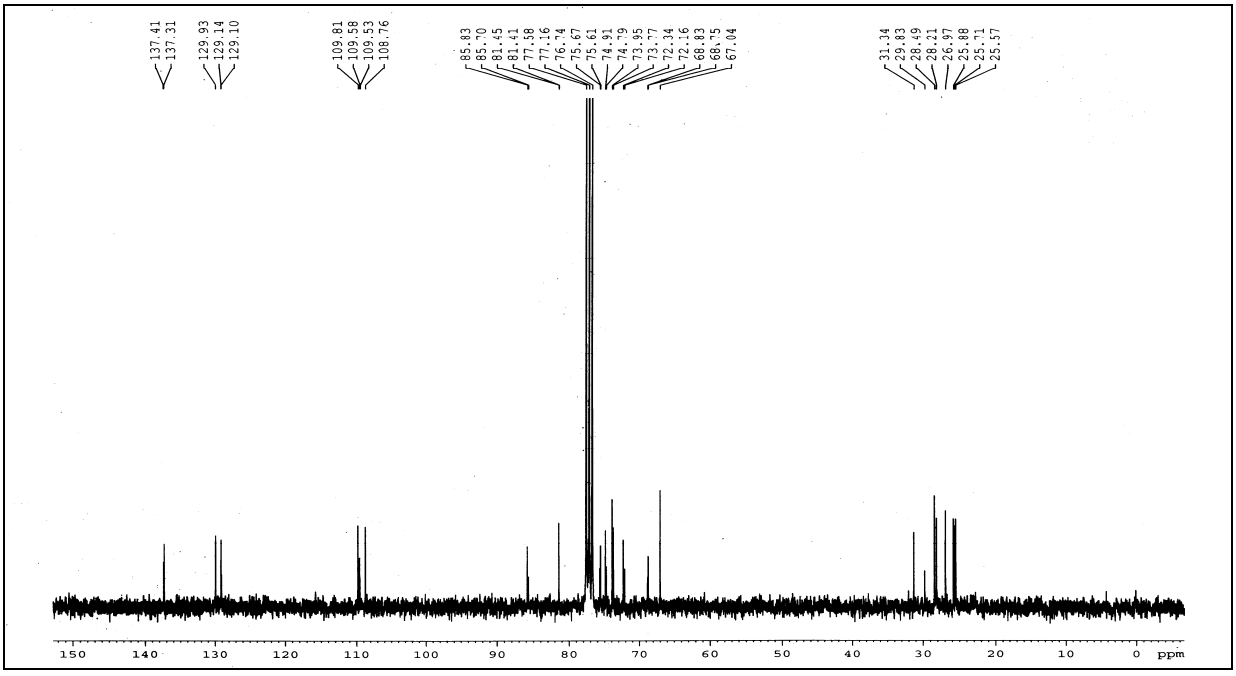
^{13}C NMR spectrum of diastereomaric mixture of 17(a-b) (75 MHz, $CDCl_3$):



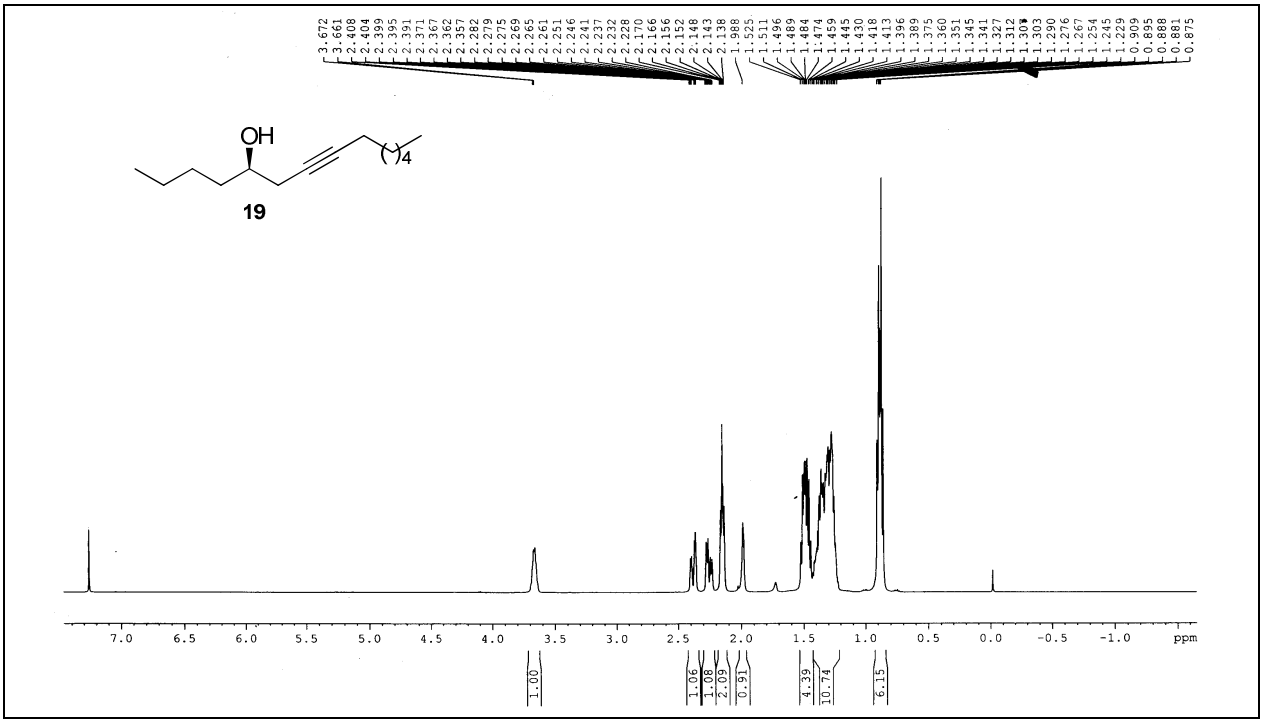
^{1}H NMR spectrum of diastereomaric mixture of 4(a-d) (300 MHz, $CDCl_3$):



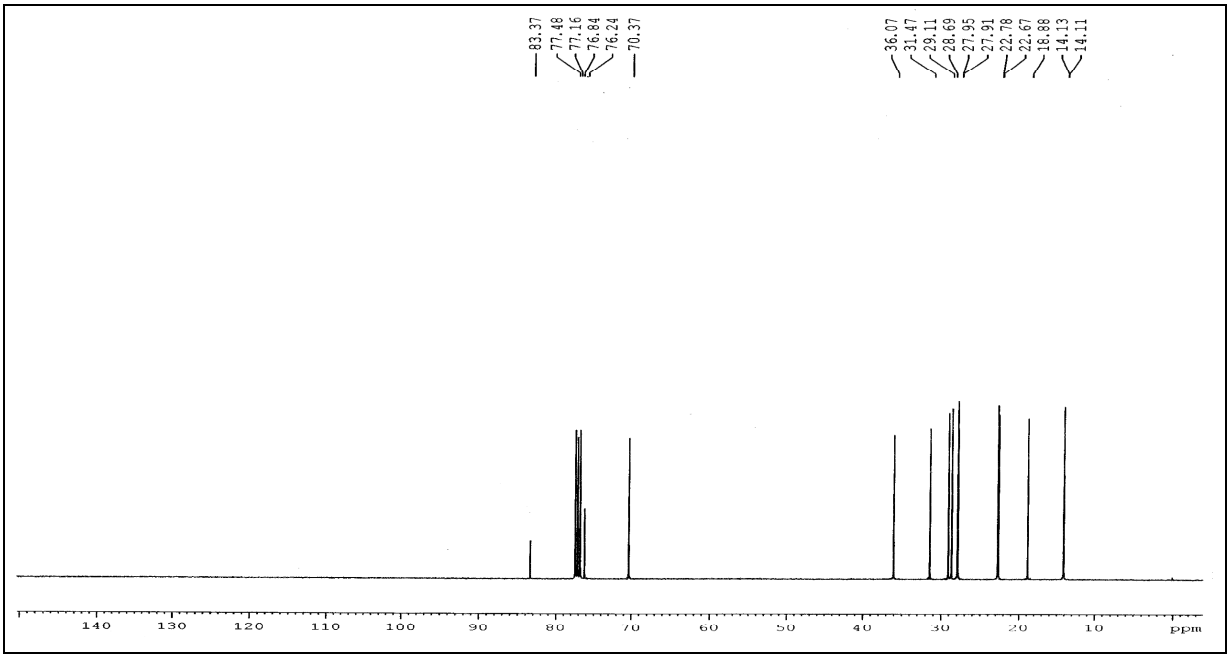
^{13}C NMR spectrum of diastereomaric mixture of 4(a-d) (75 MHz, $CDCl_3$):



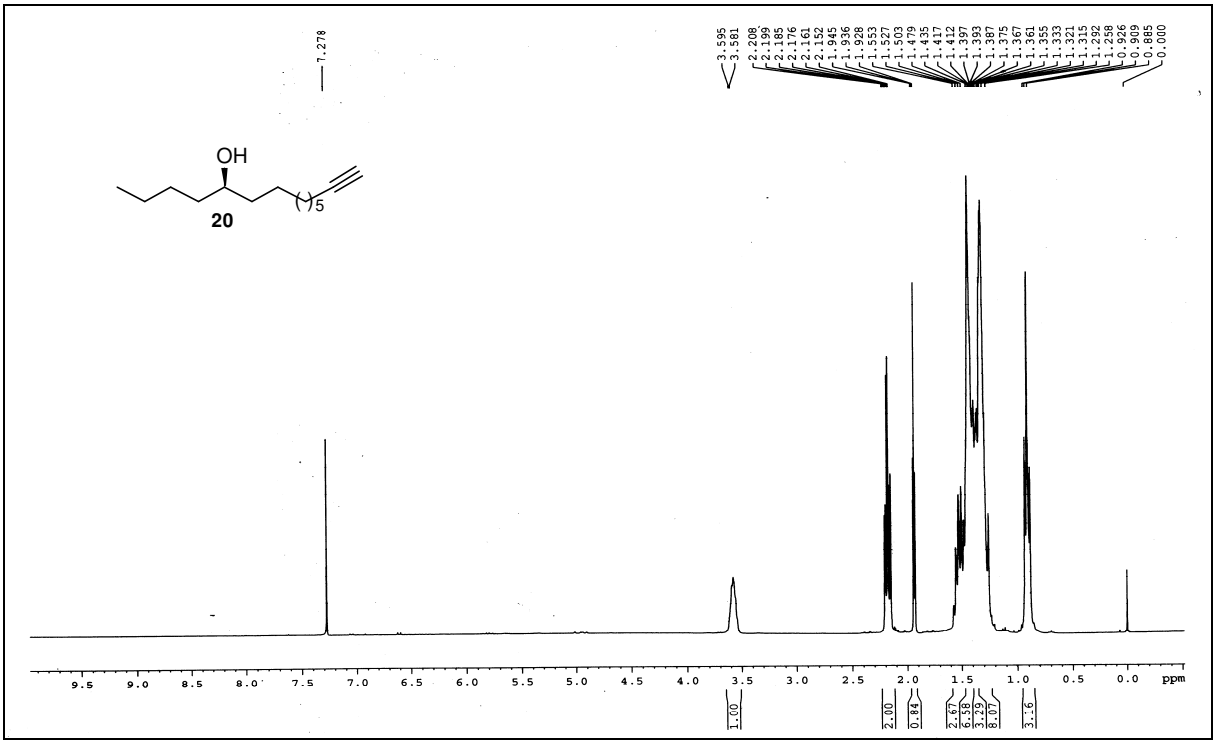
^{1}H NMR spectrum of 19 (500 MHz, $CDCl_3$):



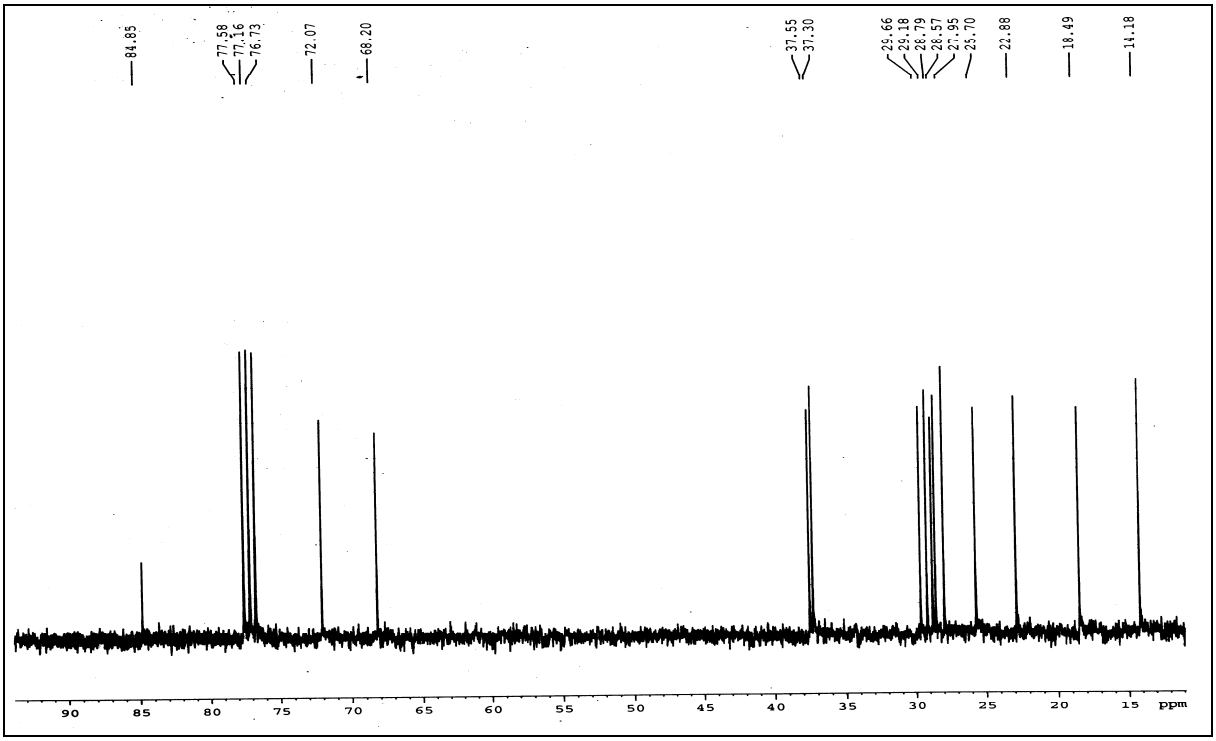
^{13}C NMR spectrum of 19 (125 MHz, $CDCl_3$):



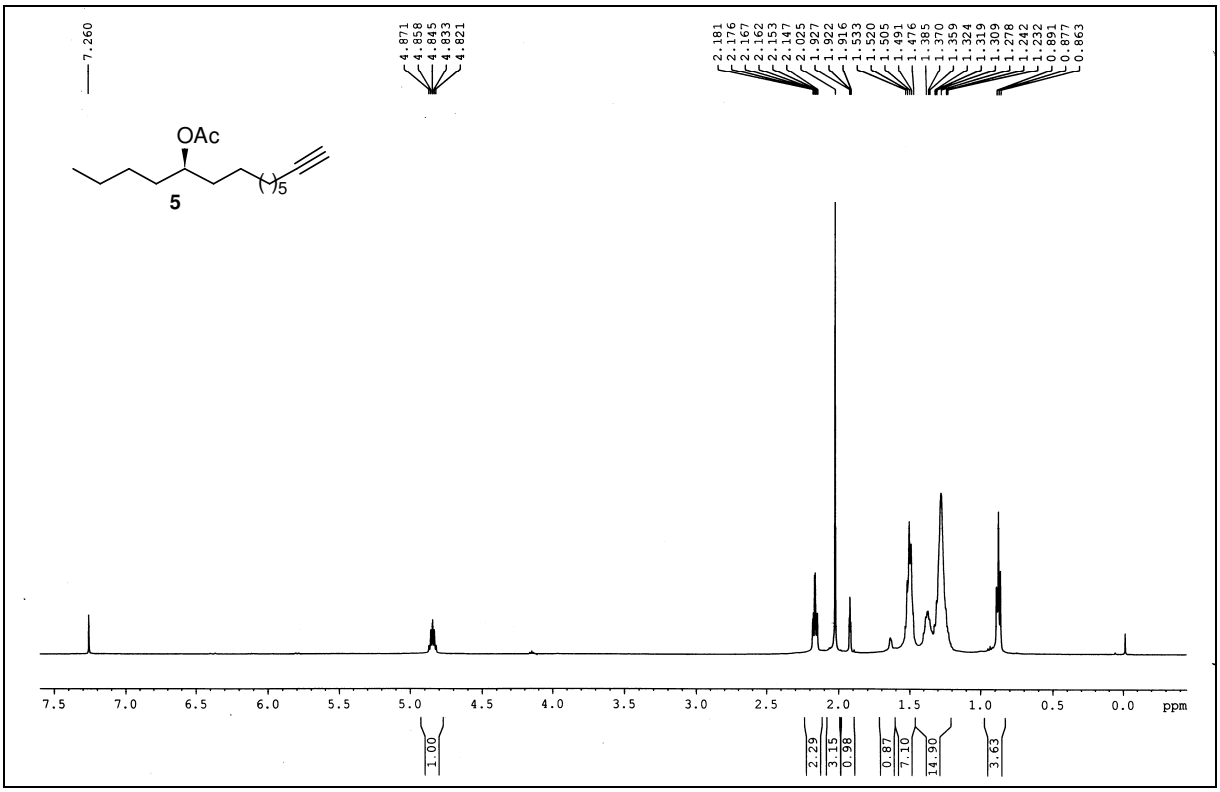
^{1}H NMR spectrum of 20 (300 MHz, $CDCl_3$):



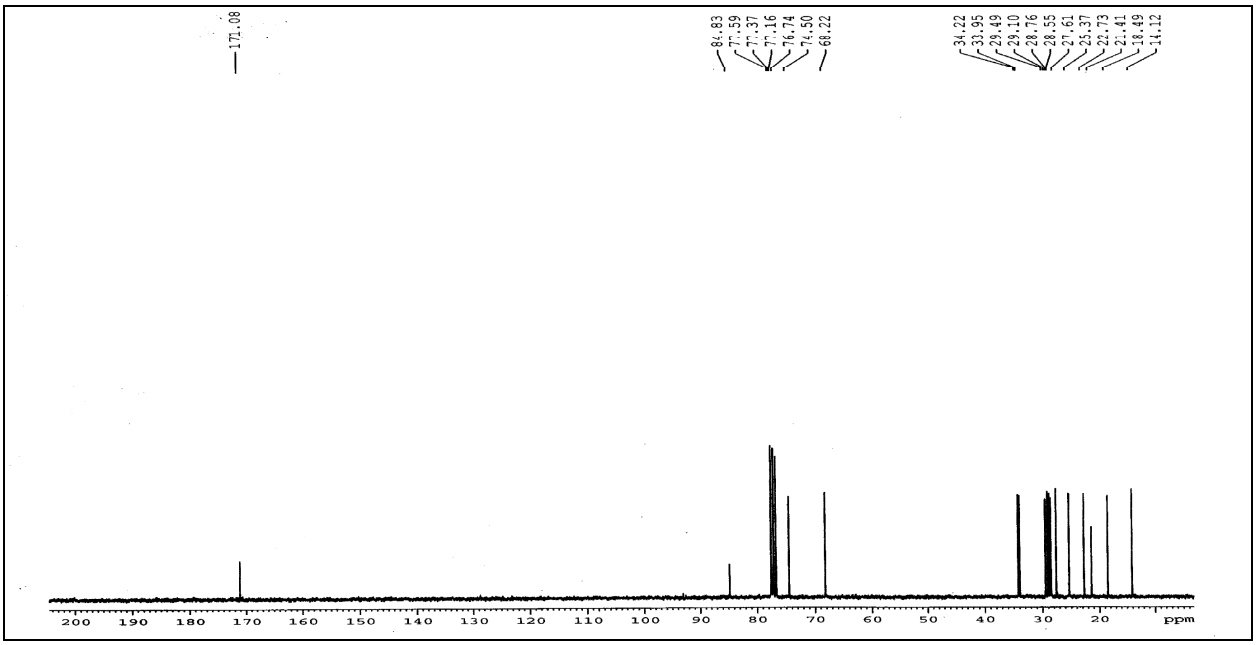
^{13}C NMR spectrum of 20 (75 MHz, $CDCl_3$):



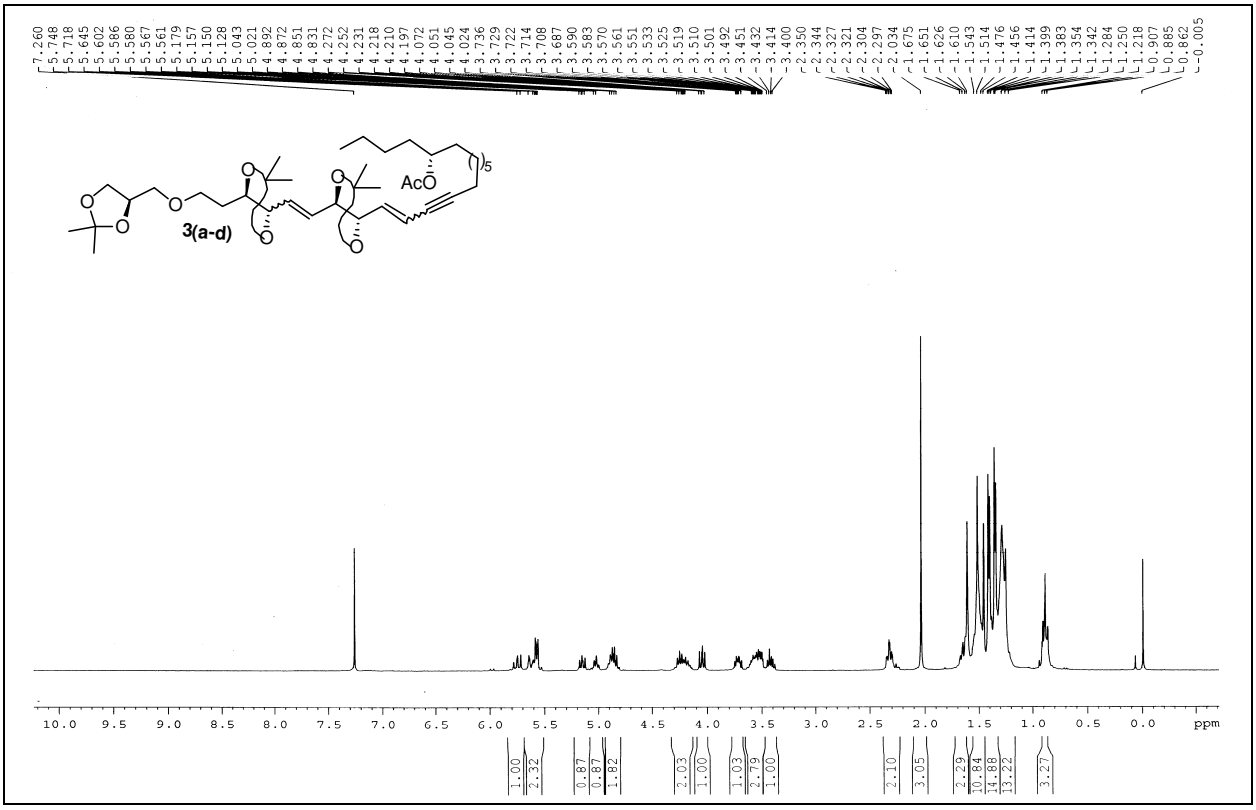
^{1}H NMR spectrum of 5 (500 MHz, $CDCl_3$):



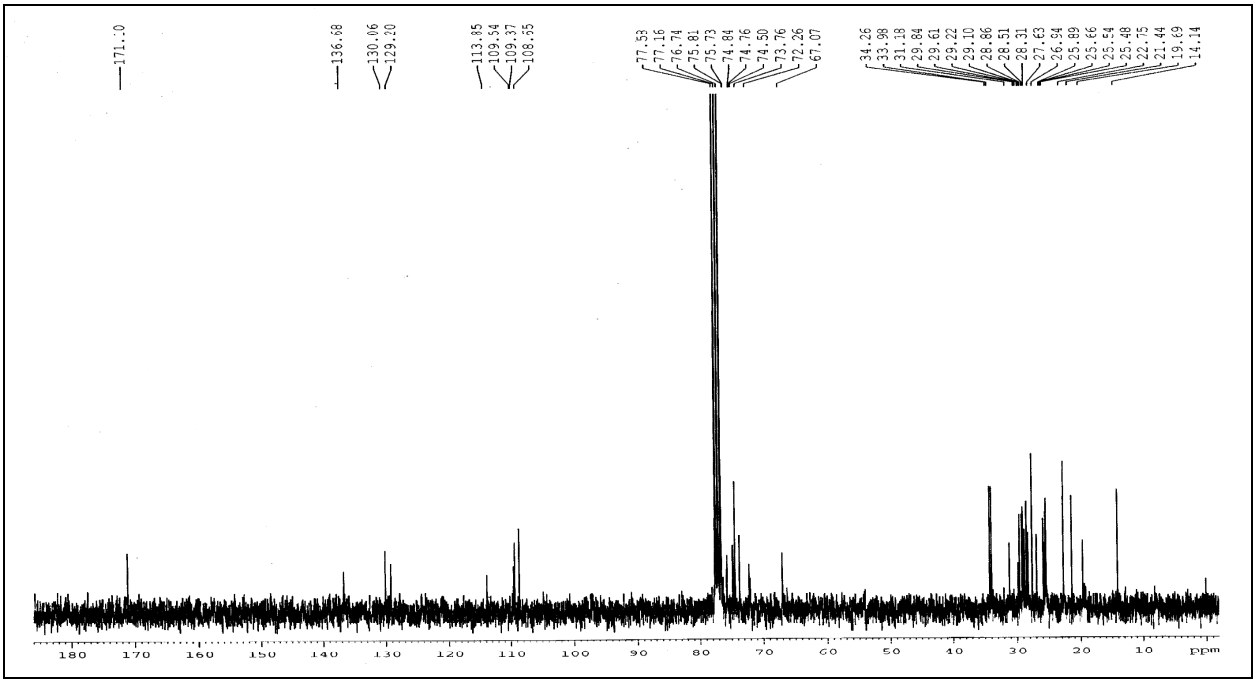
^{13}C NMR spectrum of 5 (75 MHz, $CDCl_3$):



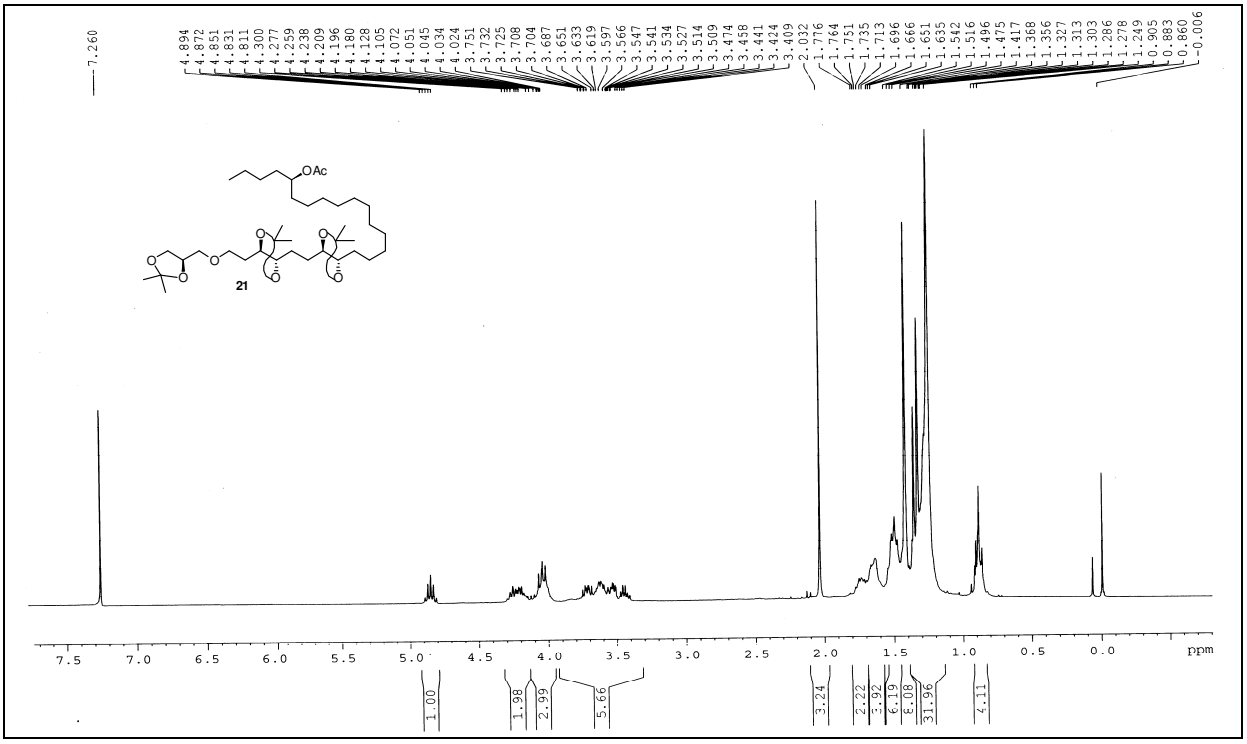
^{1}H NMR spectrum of diastereomaric mixture of 3(a-d) (300 MHz, $CDCl_3$):



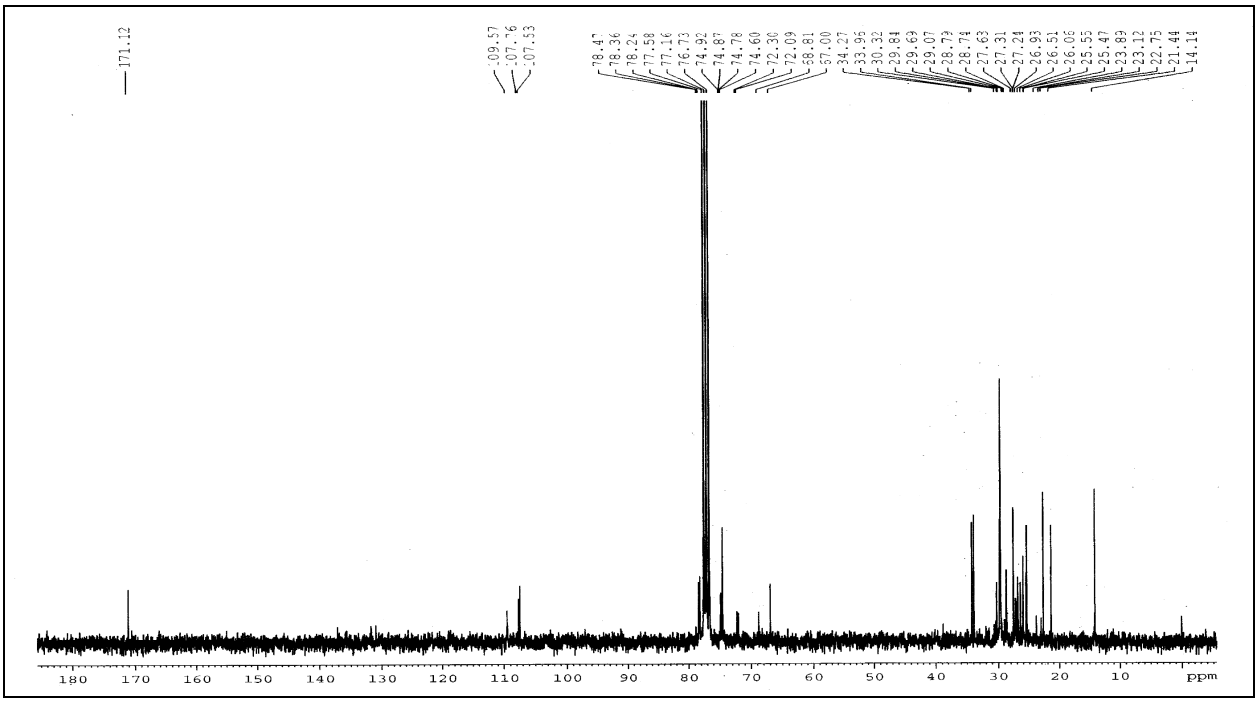
^{13}C NMR spectrum of diastereomaric mixture of 3(a-d) (75 MHz, $CDCl_3$):



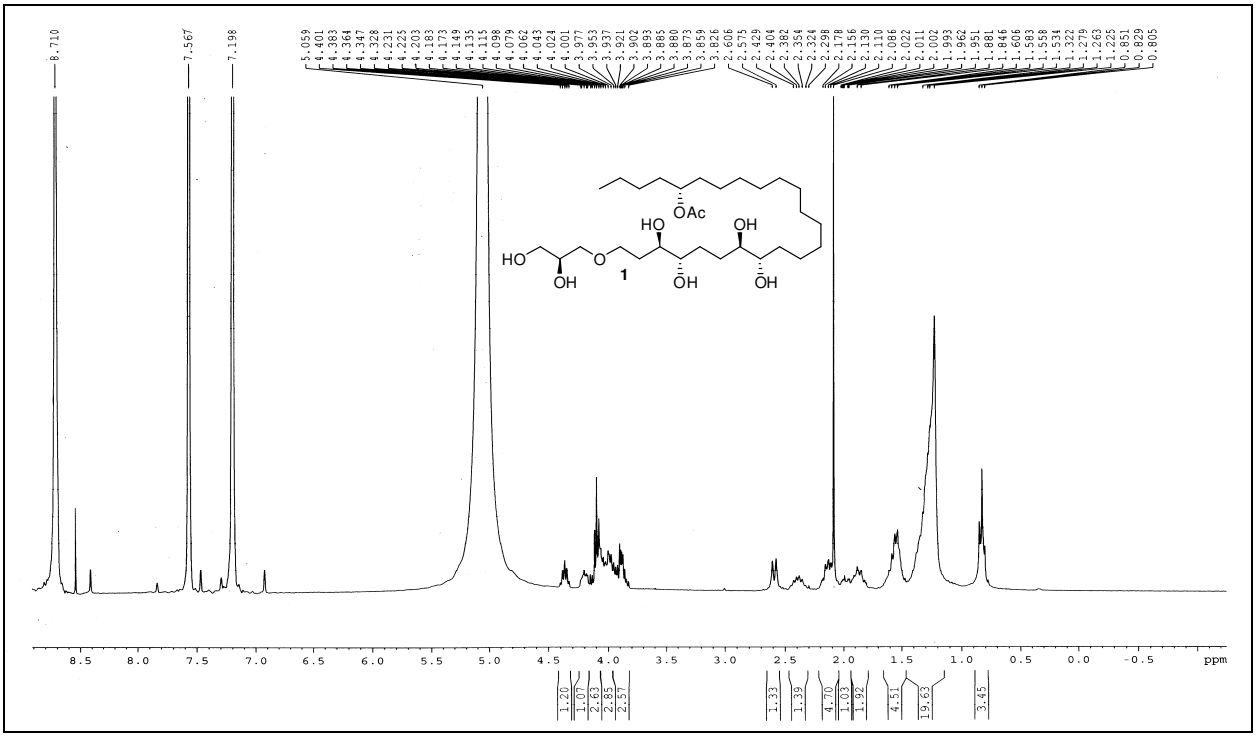
^{1}H NMR spectrum of 21 (300 MHz, $CDCl_3$):



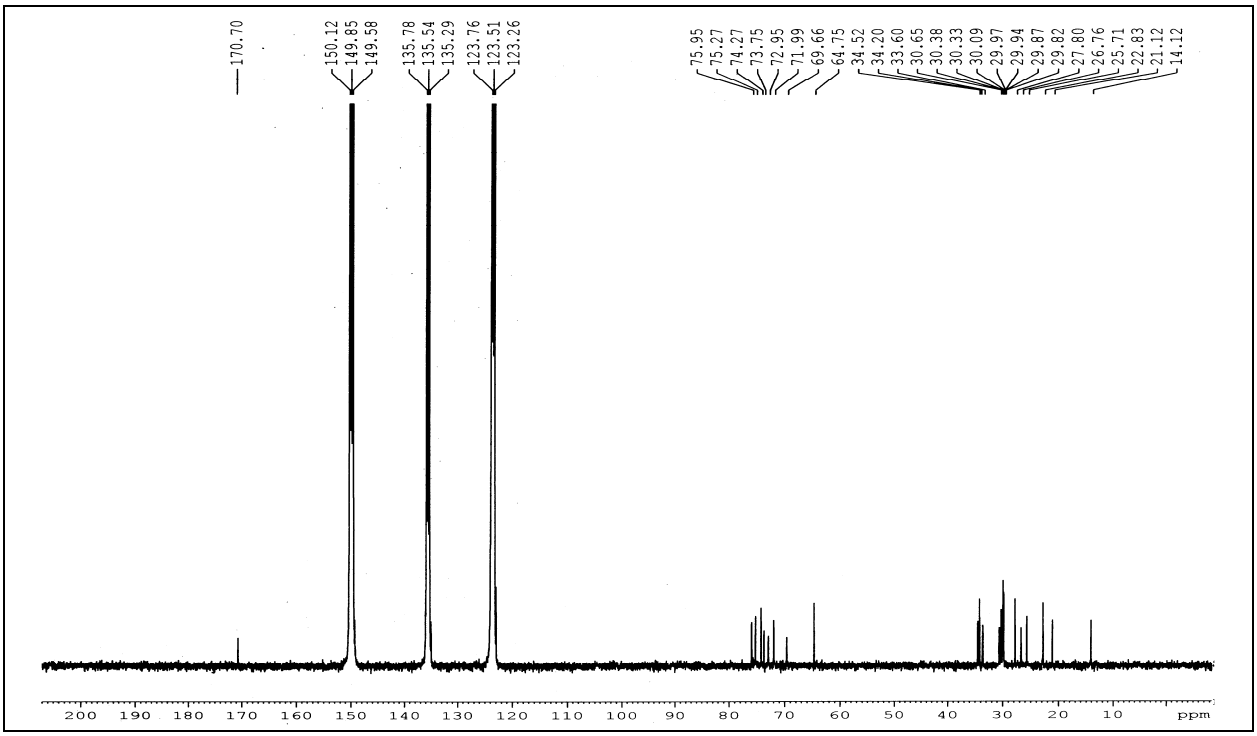
^{13}C NMR spectrum of 21 (75 MHz, $CDCl_3$):



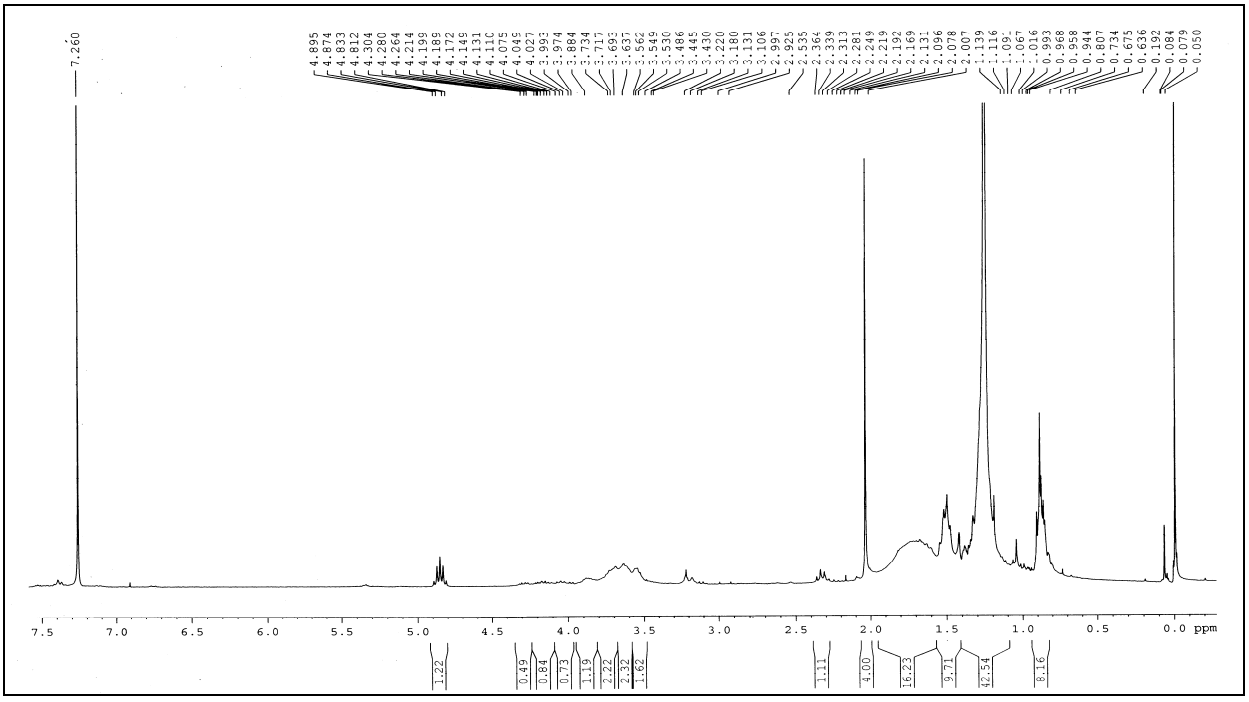
^{1}H NMR spectrum of 1 (300 MHz, C_5D_5N):



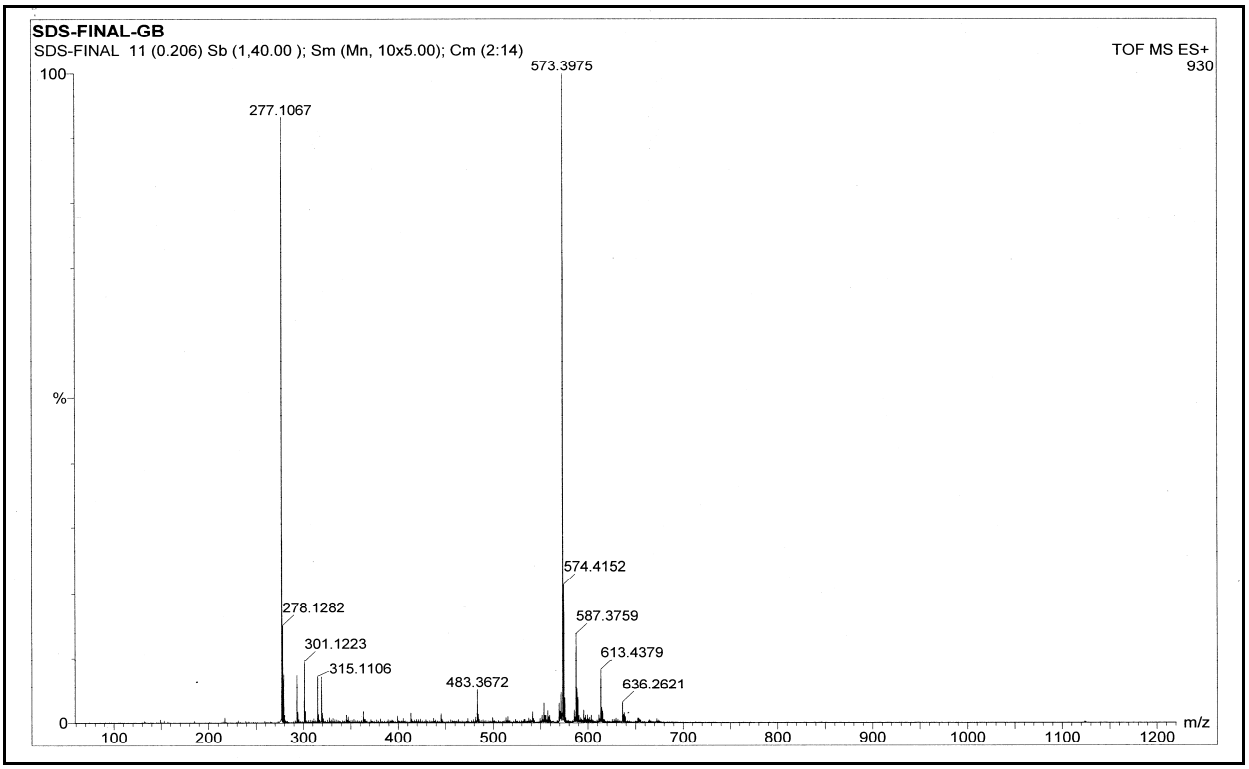
^{13}C NMR spectrum of 1 (100 MHz, C_5D_5N):



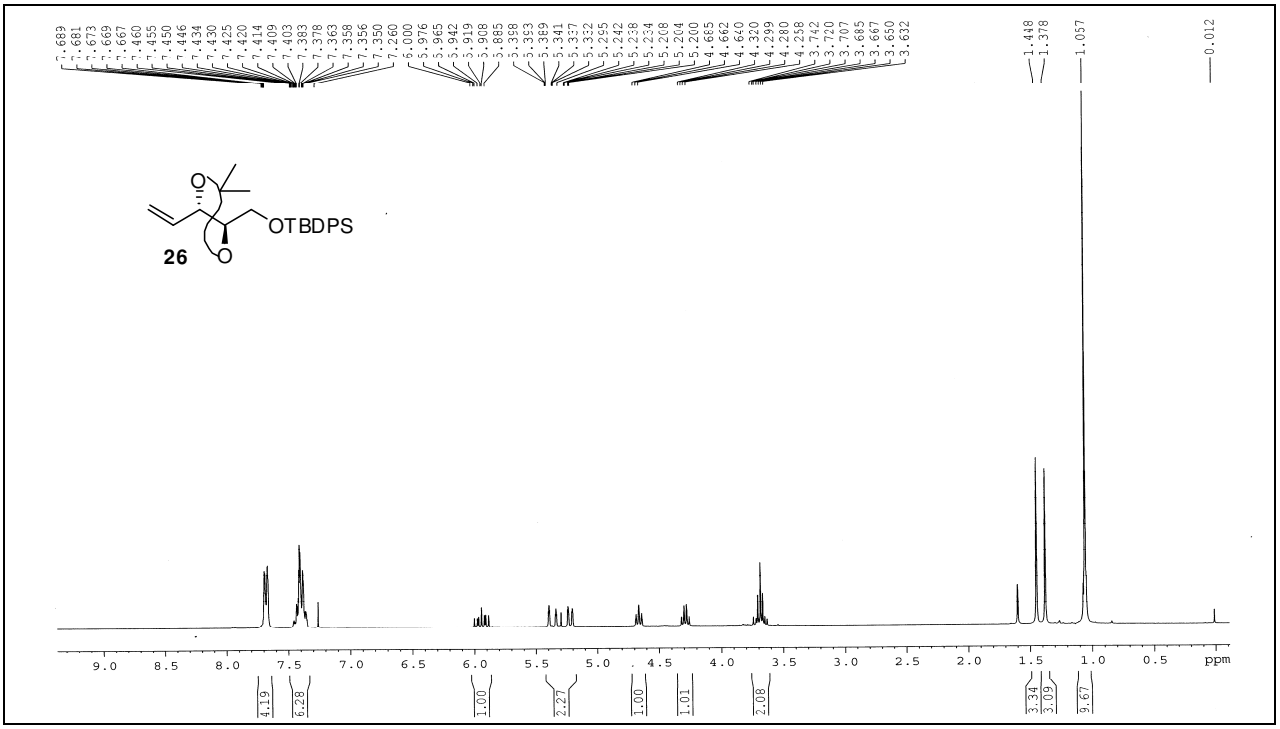
^{1}H NMR spectrum of 1 (300 MHz, $CDCl_3$):



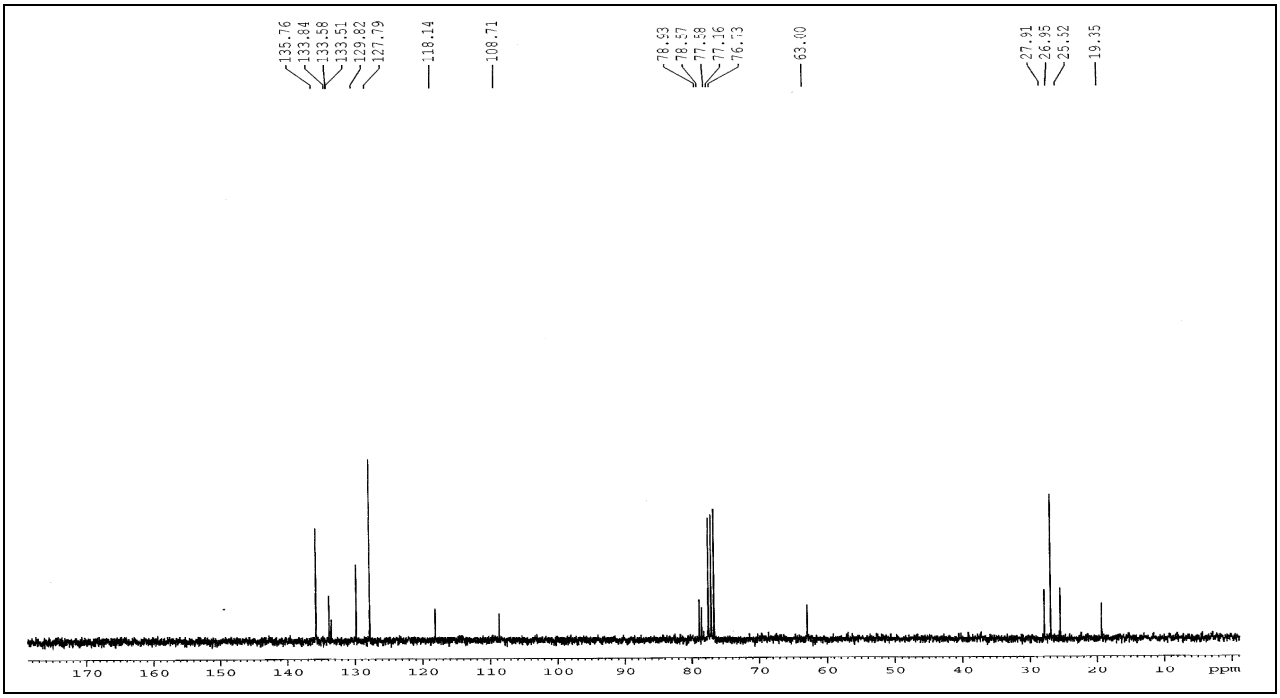
HRMS spectrum of 1:



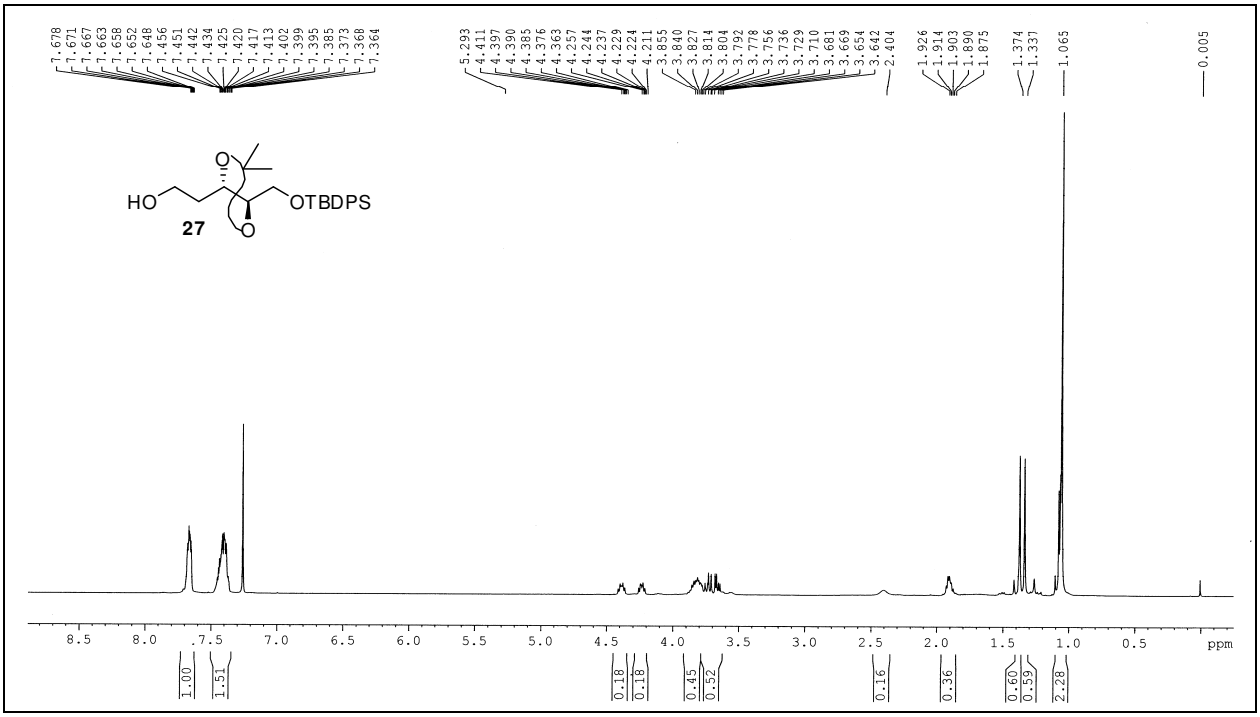
^{1}H NMR spectrum of 26 (300 MHz, $CDCl_3$):



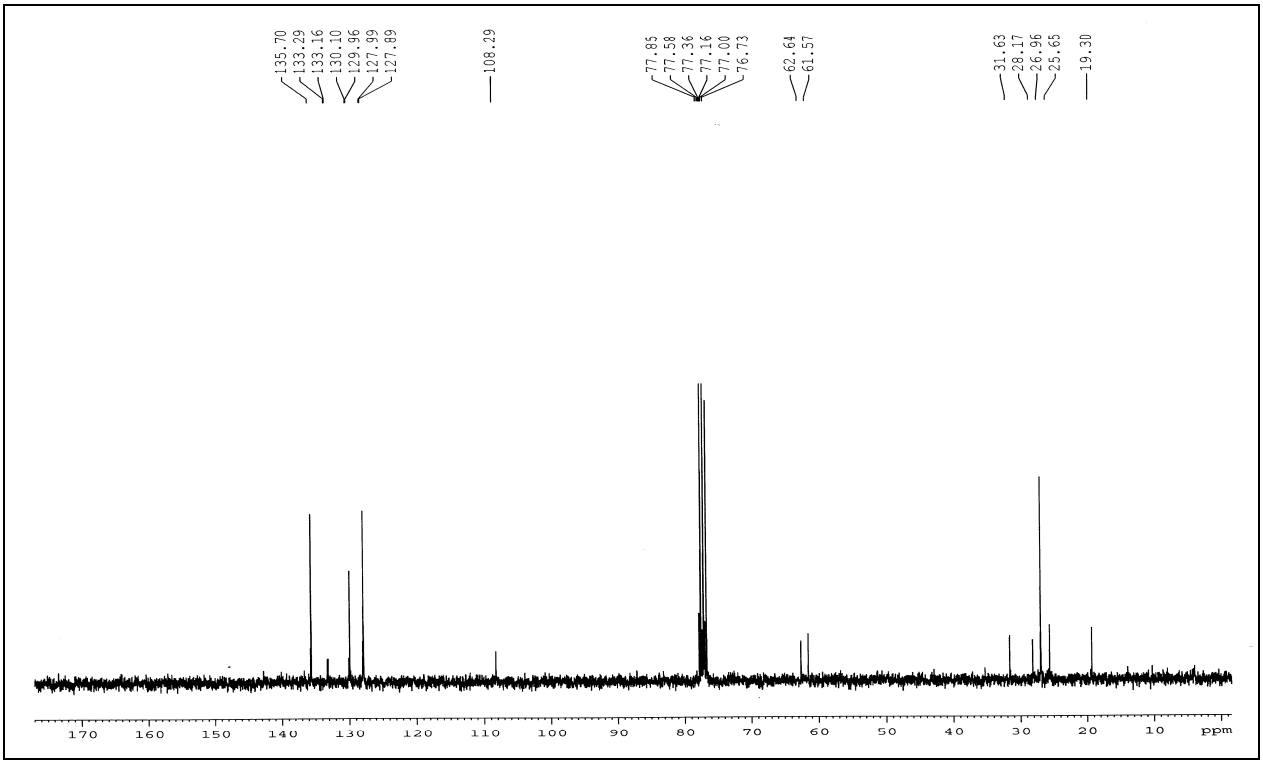
^{13}C NMR spectrum of 26 (75 MHz, $CDCl_3$):



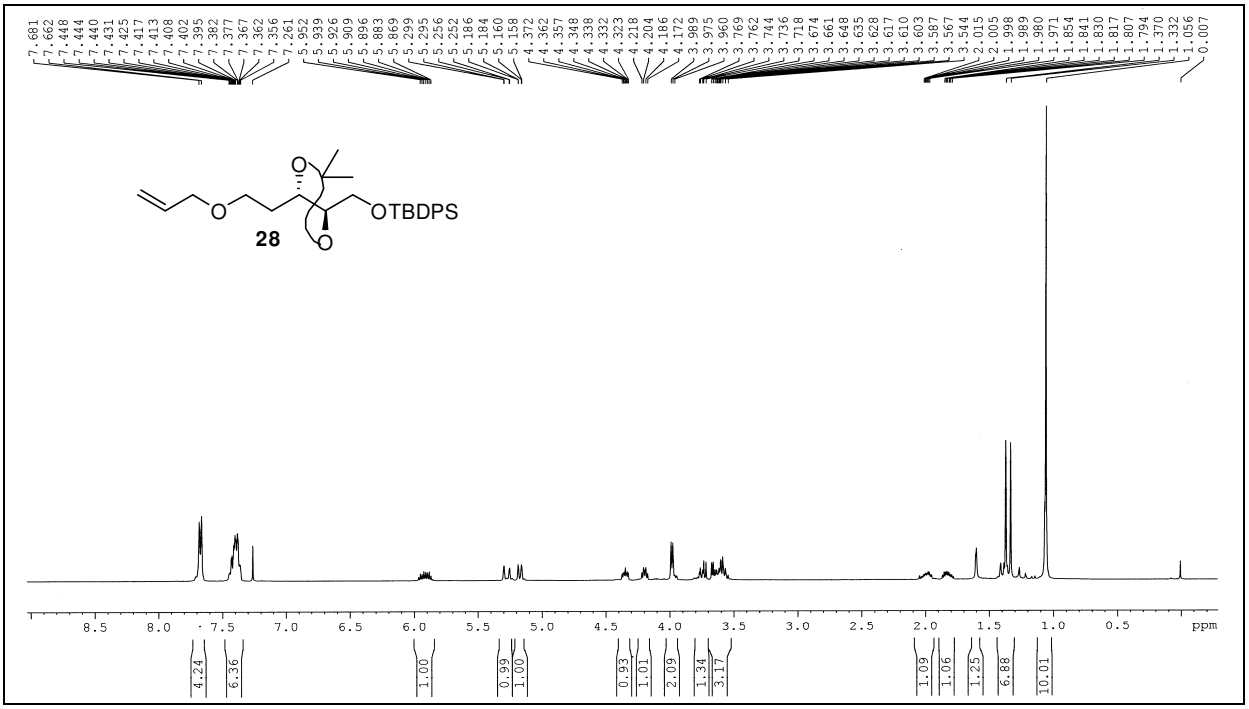
^{1}H NMR spectrum of 27 (300 MHz, $CDCl_3$):



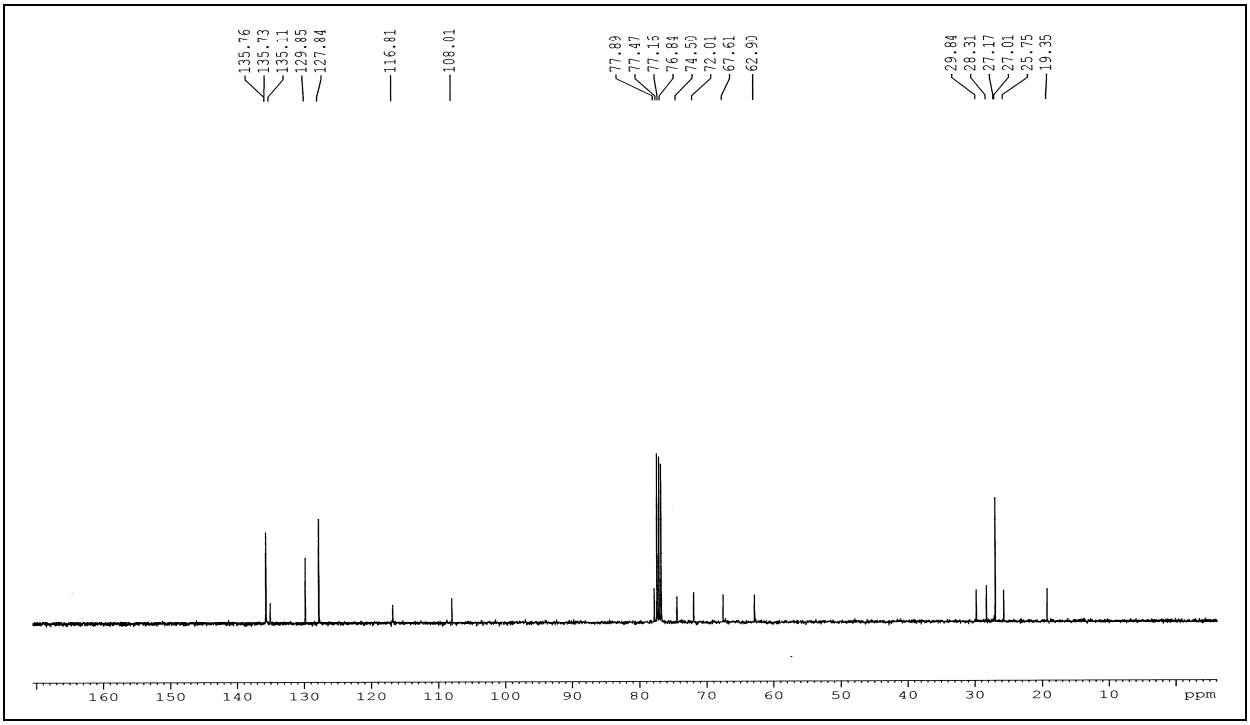
^{13}C NMR spectrum of 27 (75 MHz, $CDCl_3$):



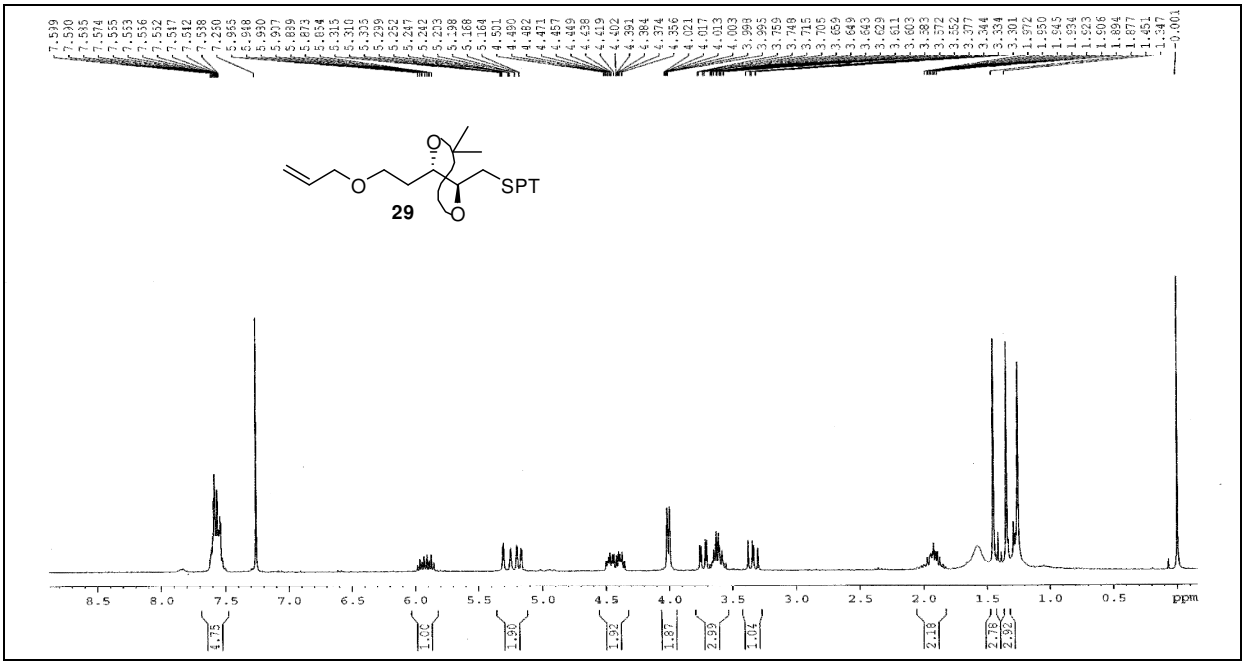
^{1}H NMR spectrum of 28 (300 MHz, $CDCl_3$):



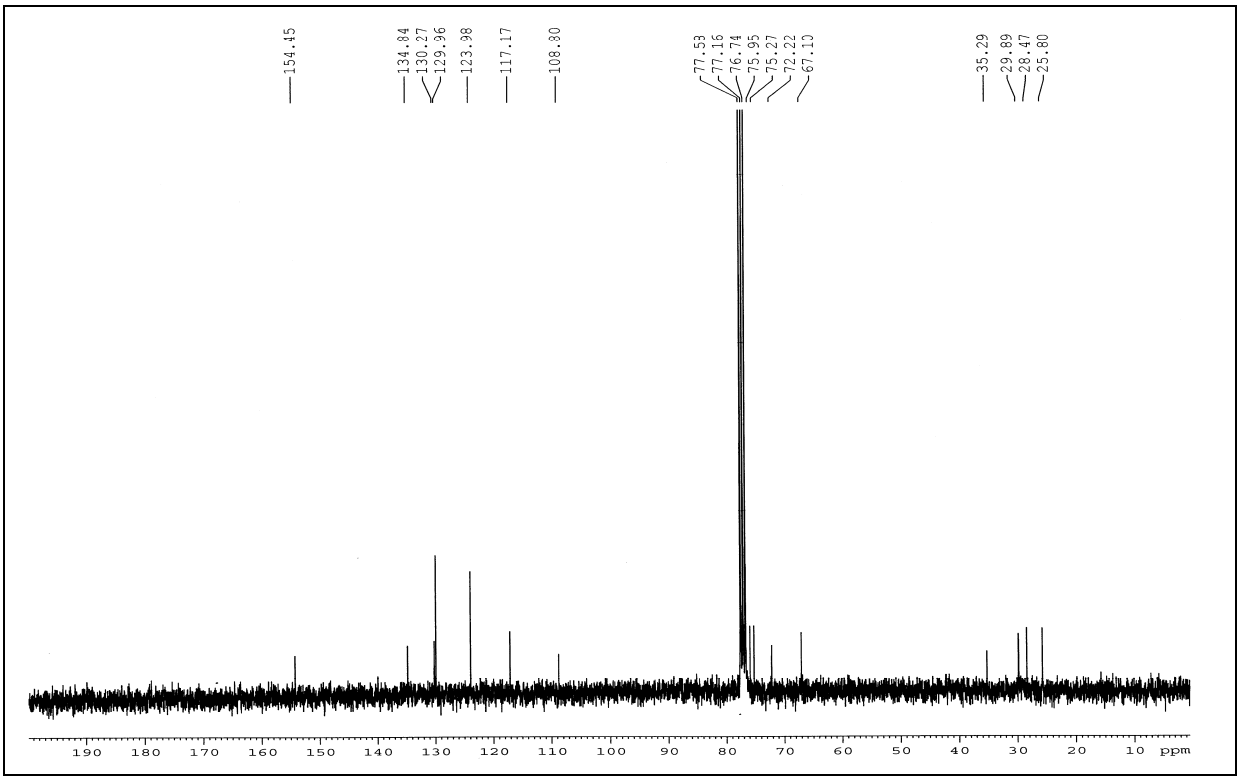
^{13}C NMR spectrum of 28 (75 MHz, $CDCl_3$):



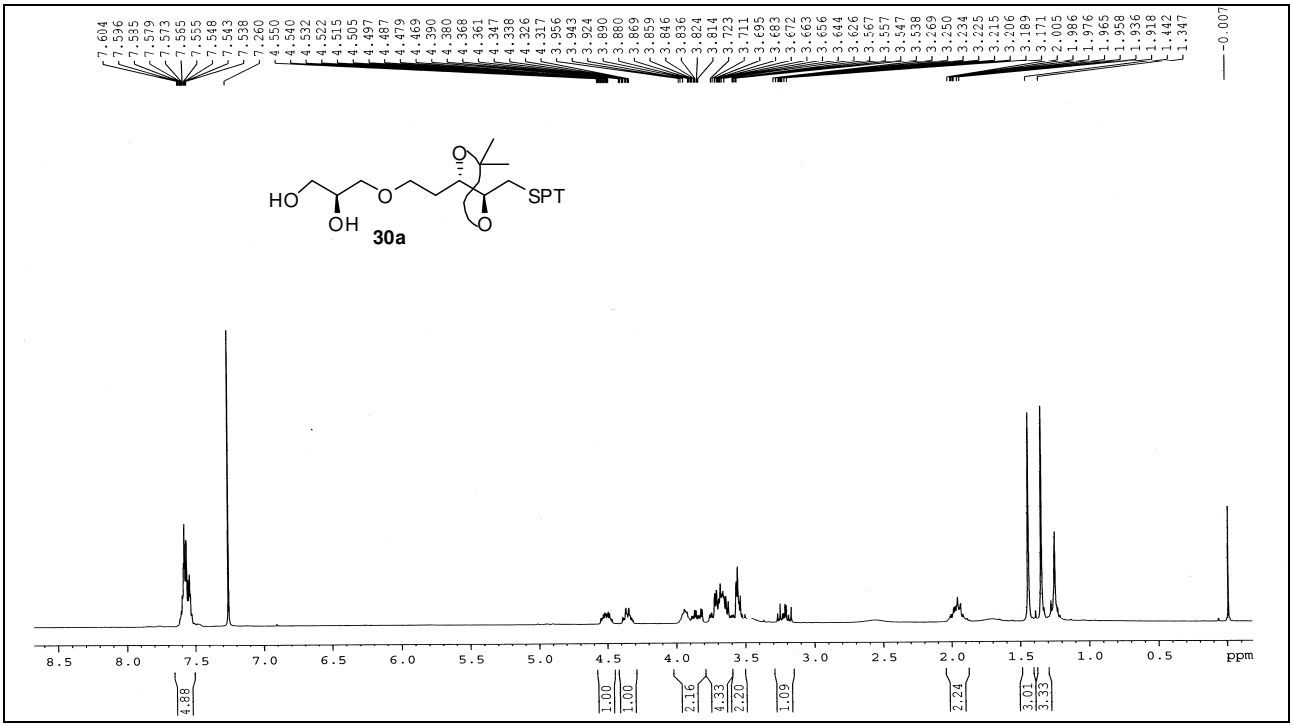
^{1}H NMR spectrum of 29 (300 MHz, $CDCl_3$):



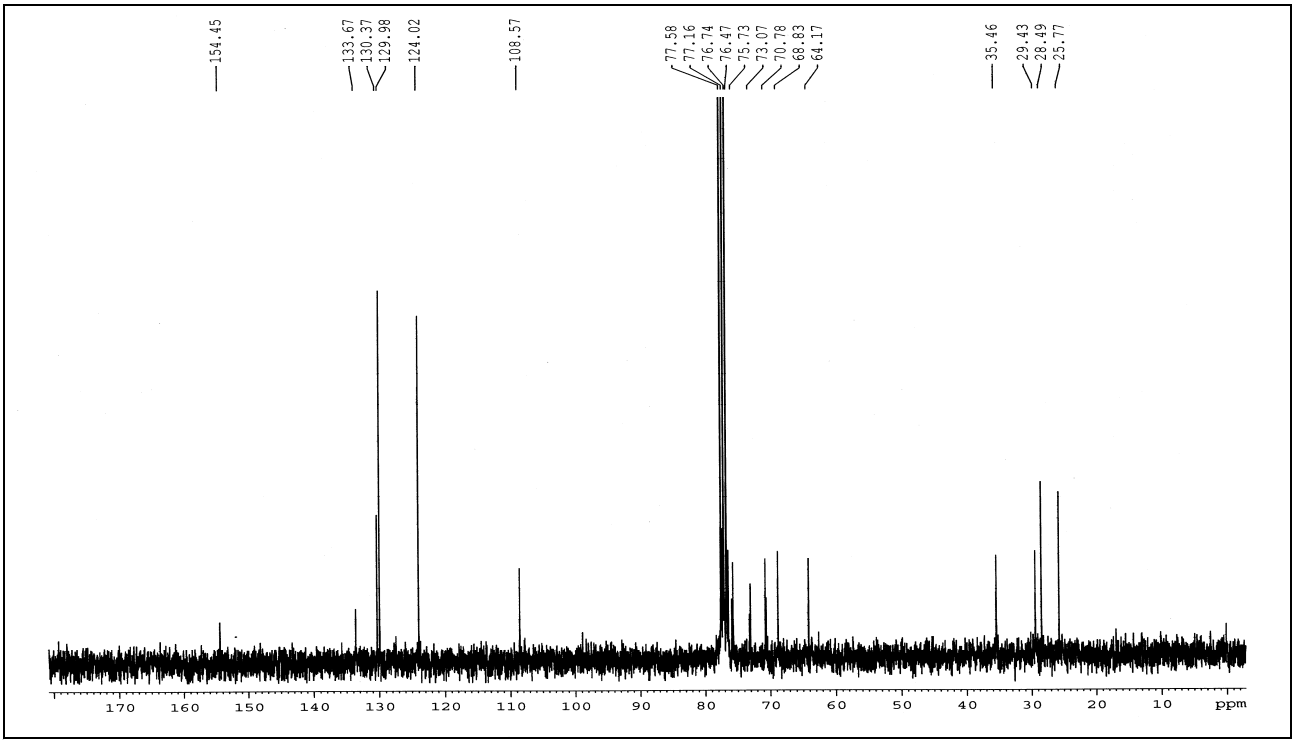
^{13}C NMR spectrum of 29 (75 MHz, $CDCl_3$):



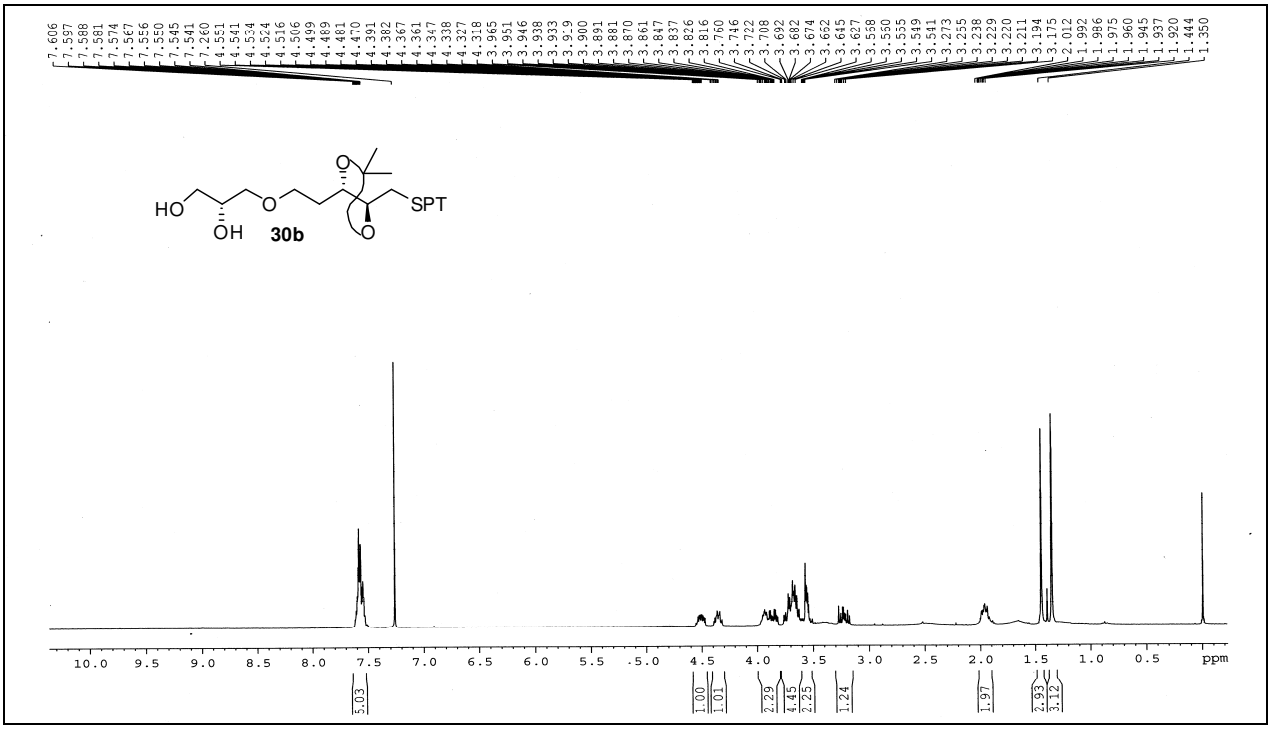
^{1}H NMR spectrum of 30a (300 MHz, $CDCl_3$):



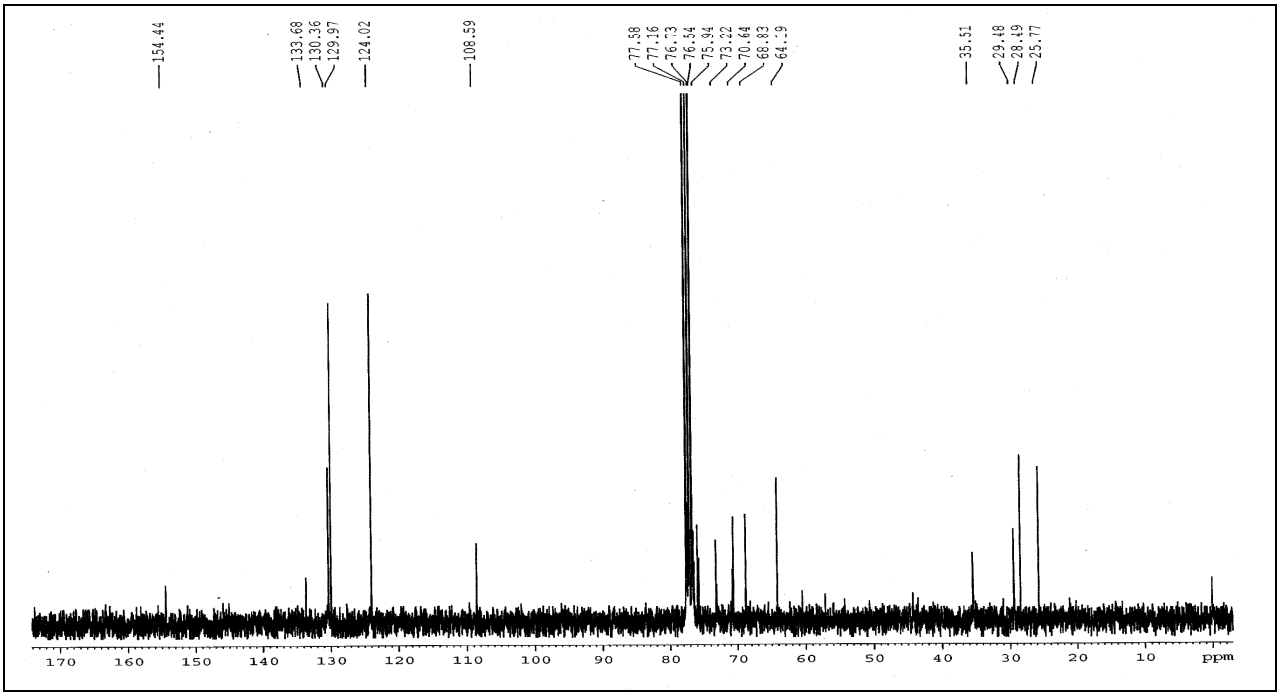
^{13}C NMR spectrum of 30a (75 MHz, $CDCl_3$):



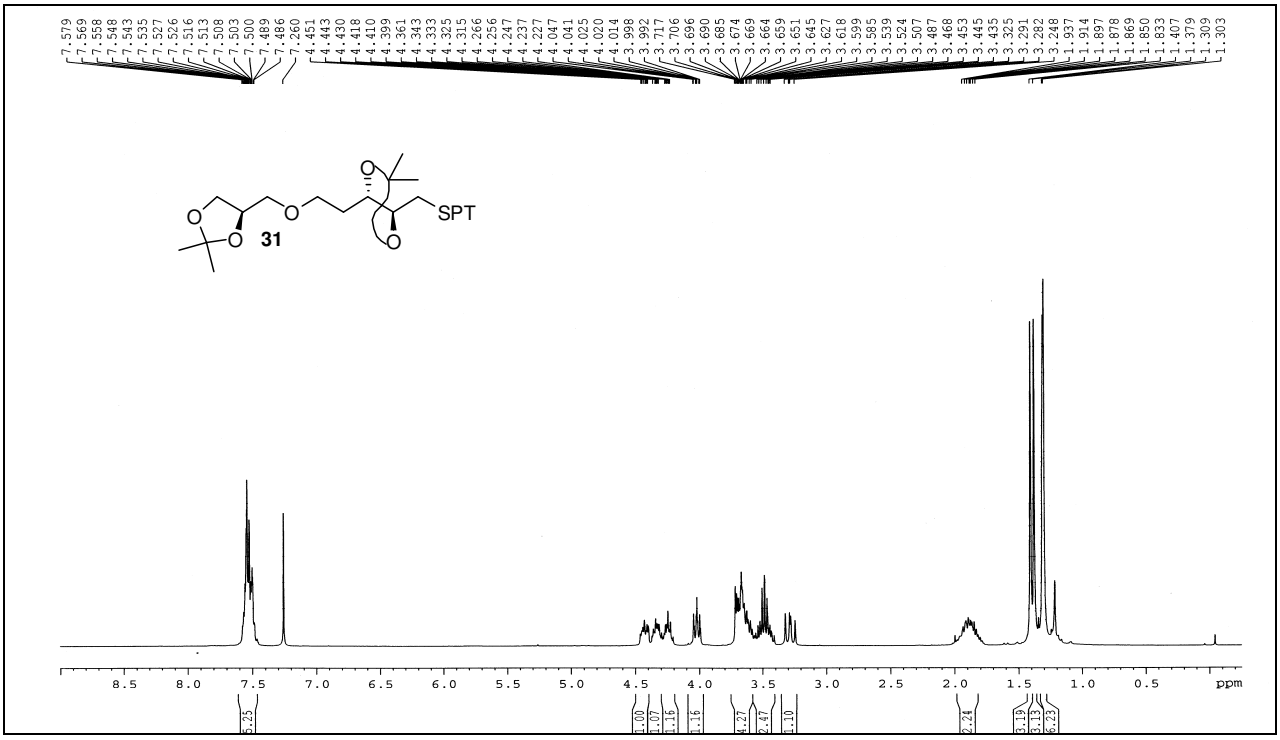
^{1}H NMR spectrum of 30b (300 MHz, $CDCl_3$):



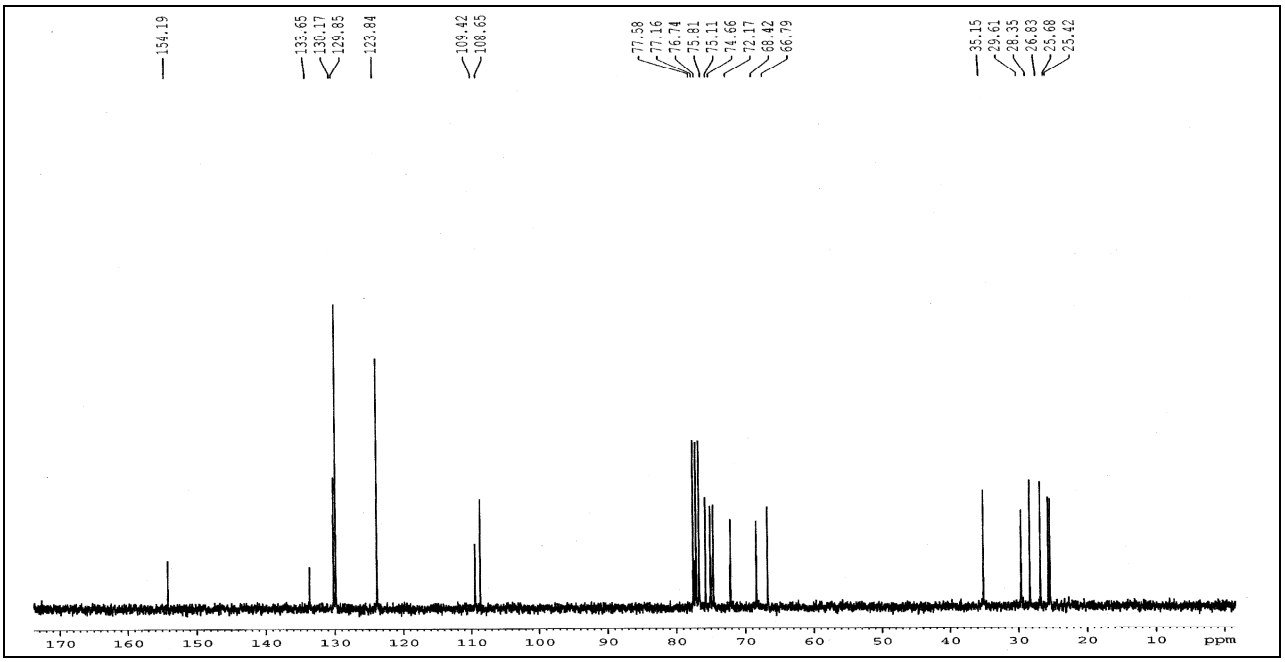
^{13}C NMR spectrum of 30b (75 MHz, $CDCl_3$):



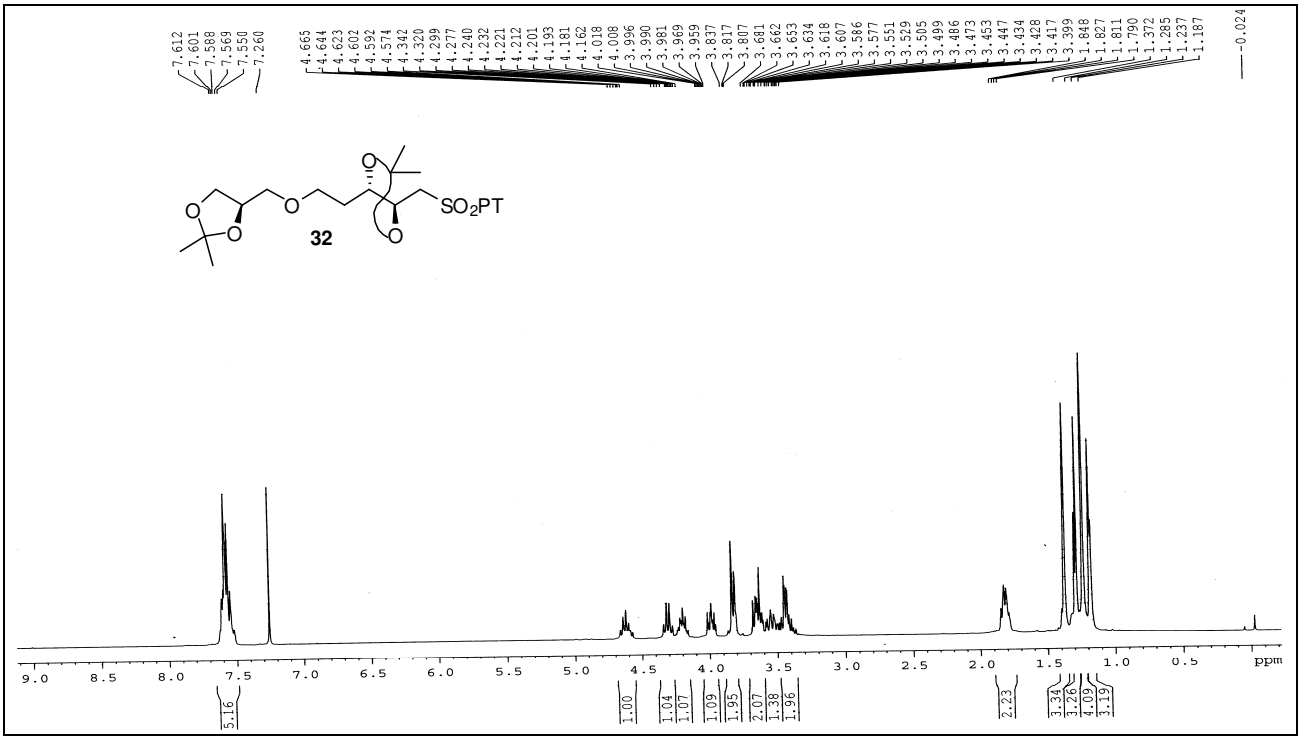
^{1}H NMR spectrum of 31 (300 MHz, $CDCl_3$):



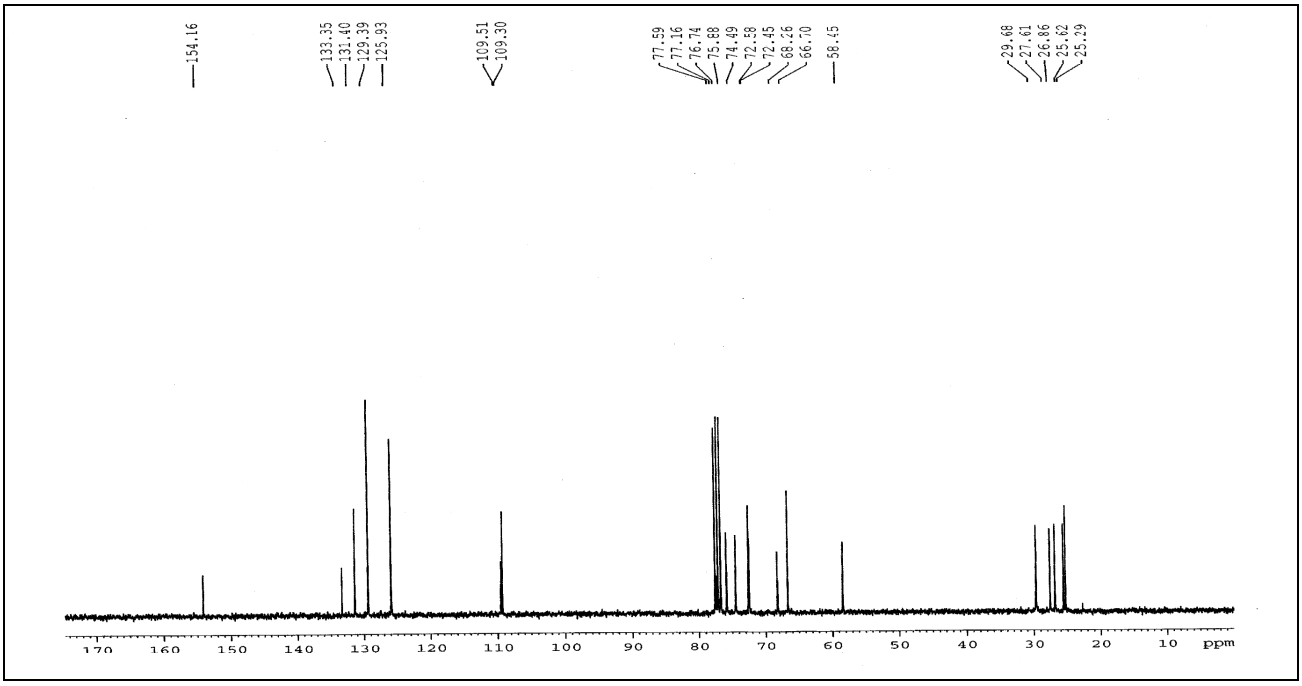
^{13}C NMR spectrum of 31 (75 MHz, $CDCl_3$):



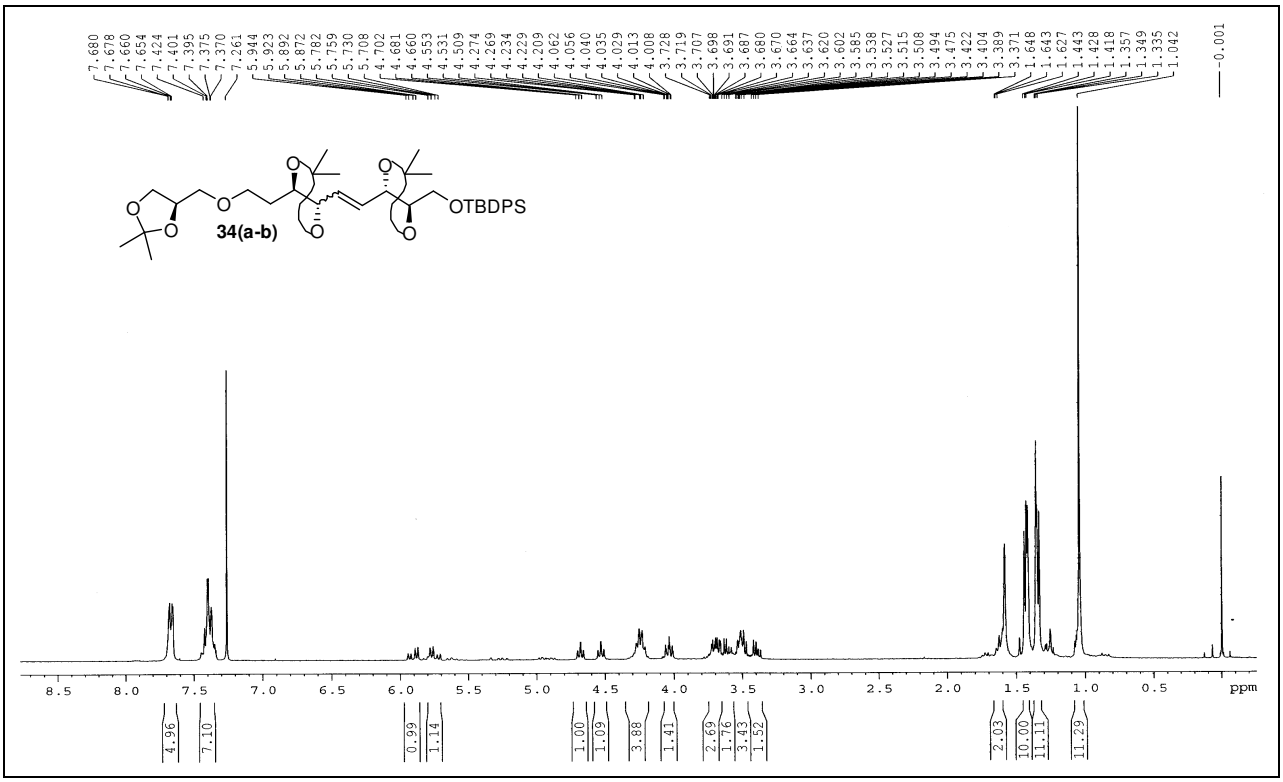
^{1}H NMR spectrum of 32 (300 MHz, $CDCl_3$):



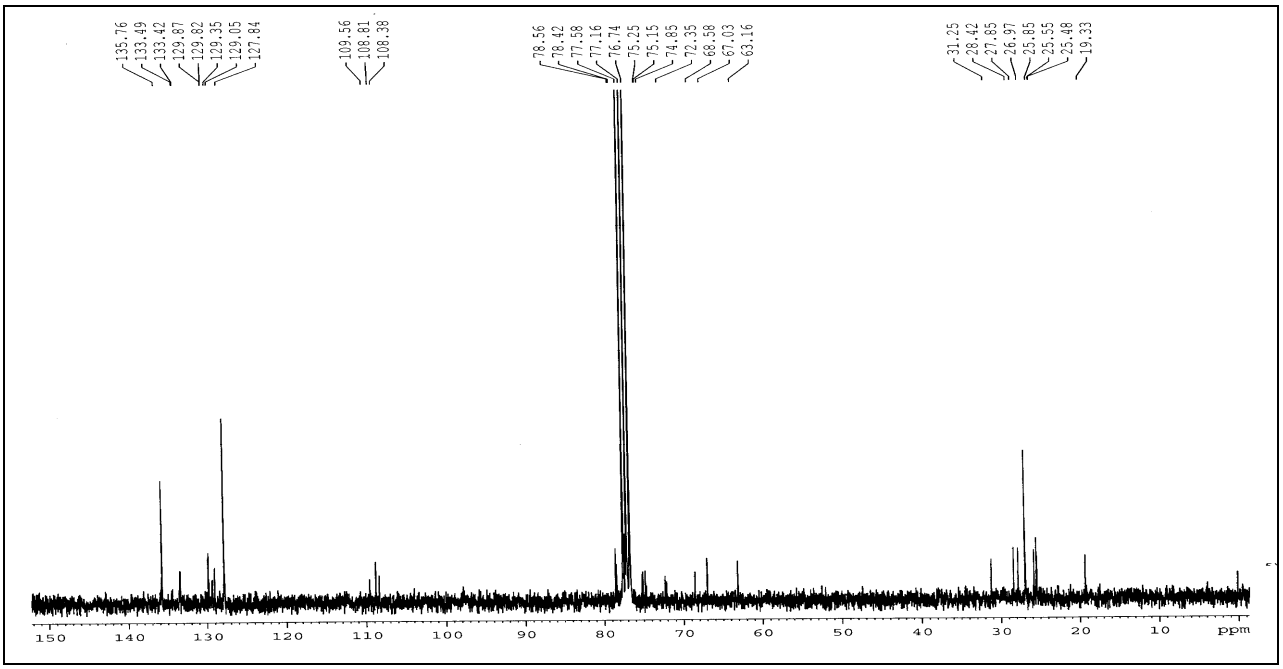
^{13}C NMR spectrum of 32 (75 MHz, $CDCl_3$):



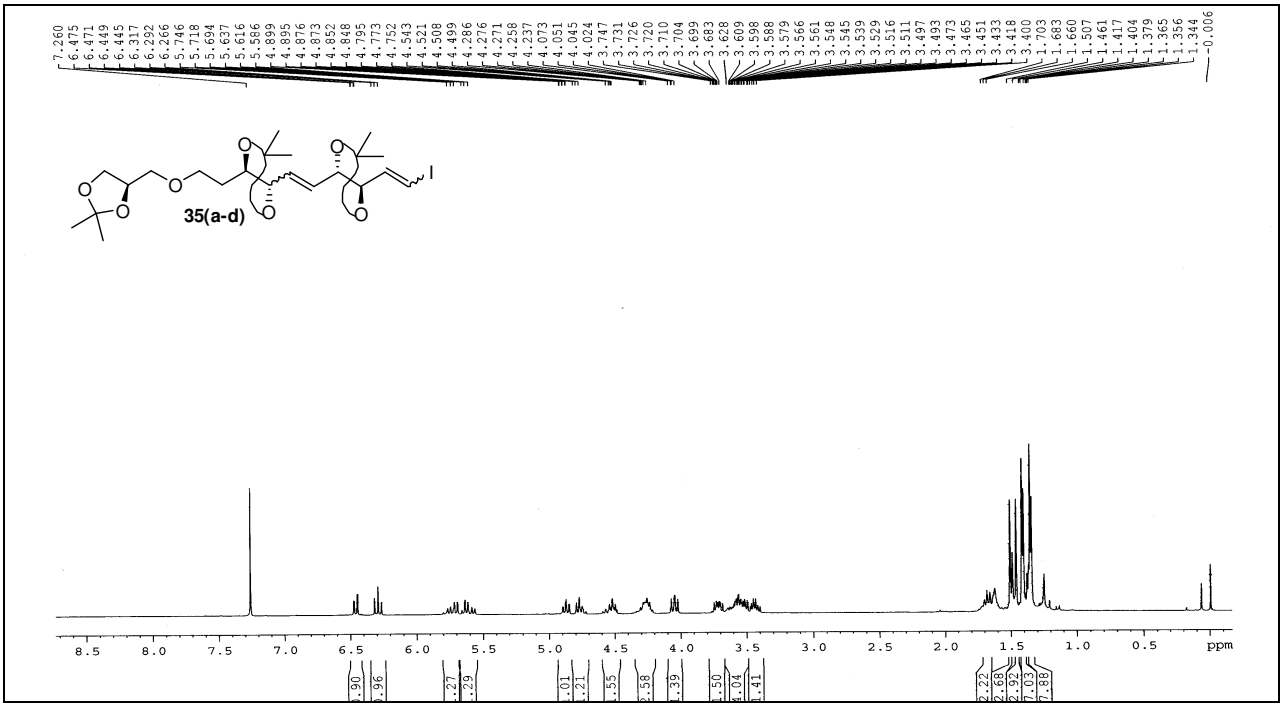
^{1}H NMR spectrum of 34(a-b) (300 MHz, $CDCl_3$):



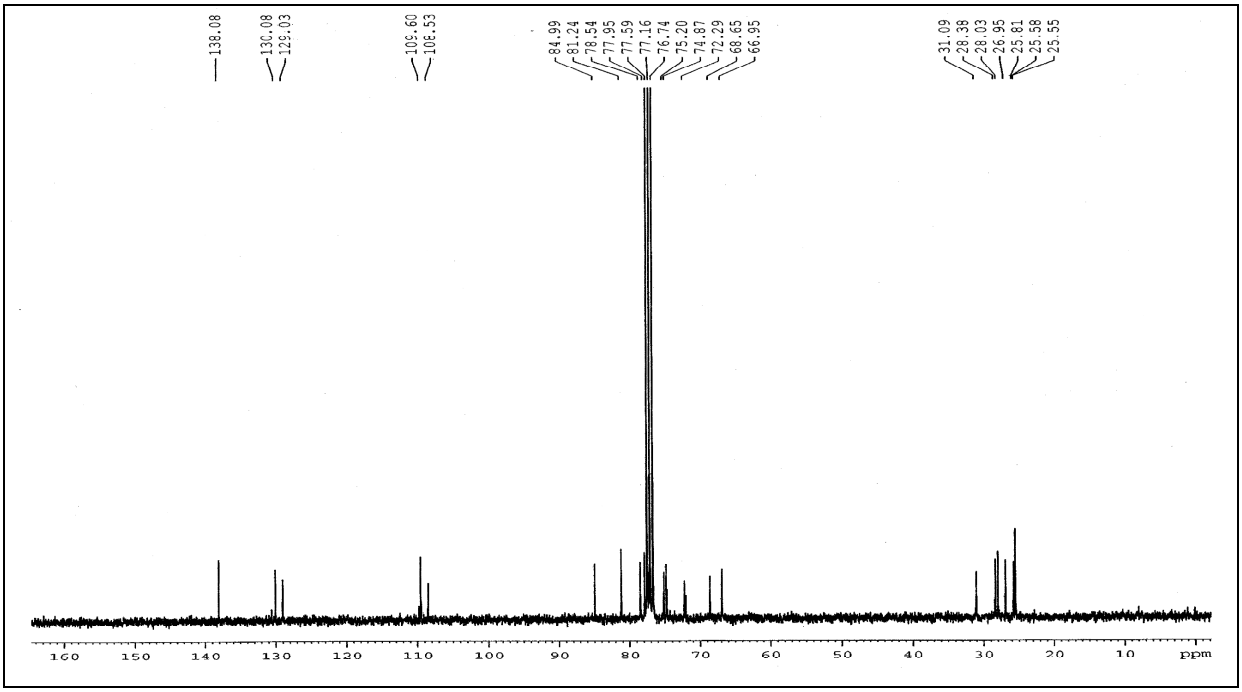
^{13}C NMR spectrum of 34(a-b) (75 MHz, $CDCl_3$):



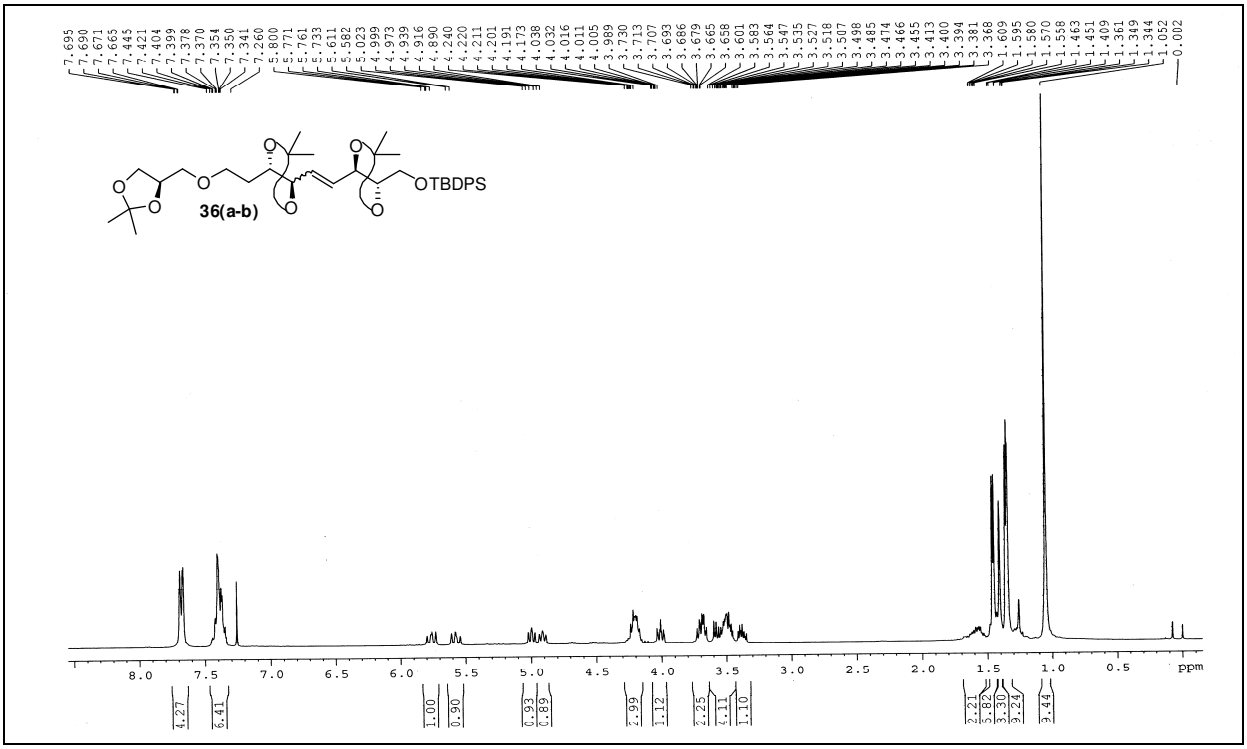
^{1}H NMR spectrum of 35(a-d) (300 MHz, $CDCl_3$):



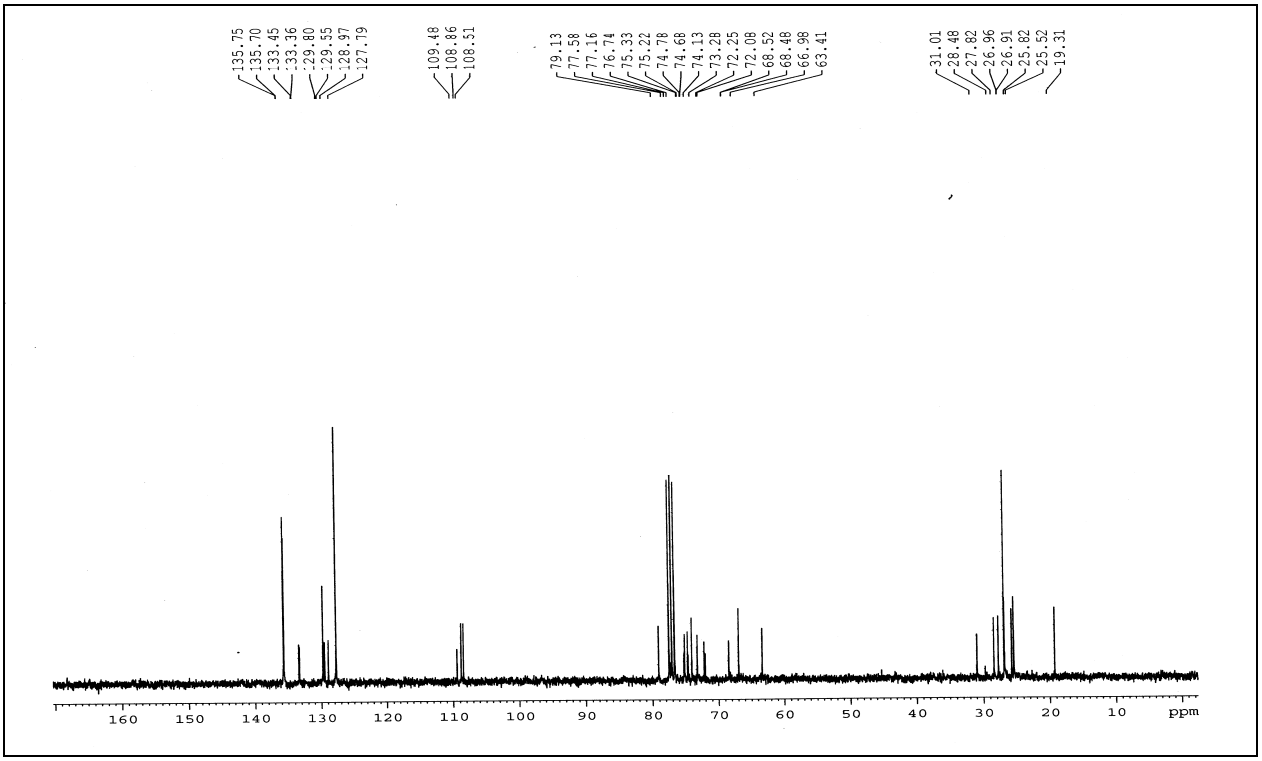
^{13}C NMR spectrum of 35(a-d) (75 MHz, $CDCl_3$):



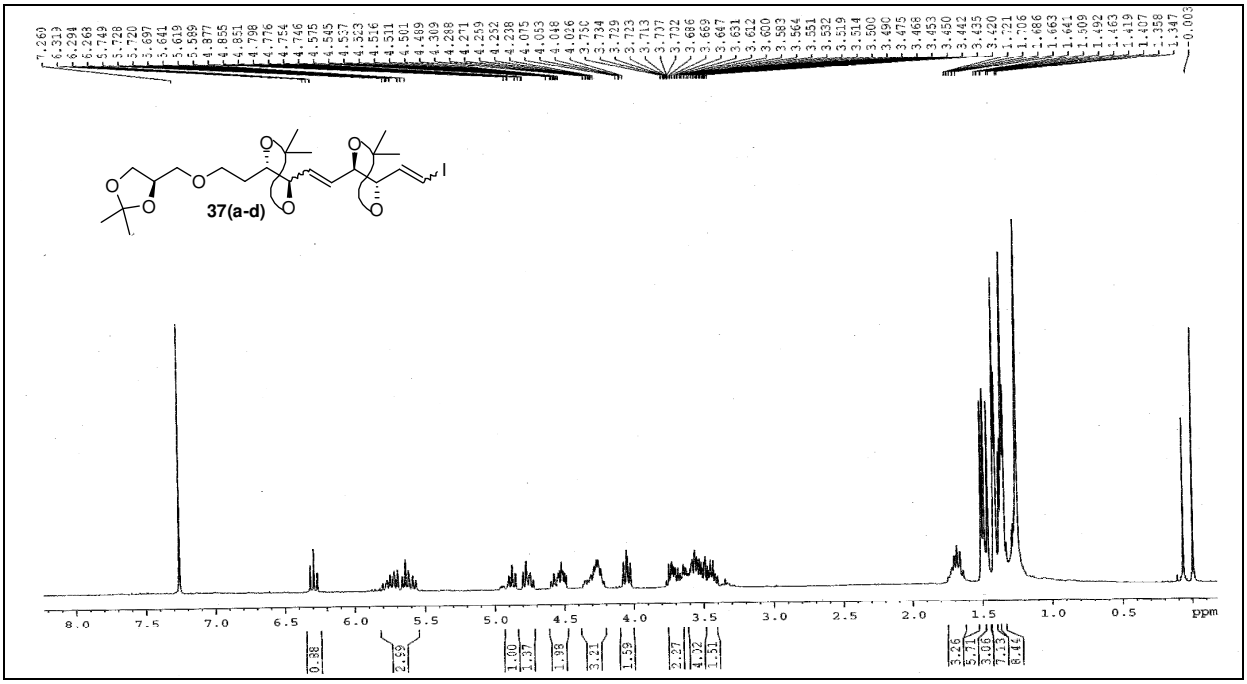
^{1}H NMR spectrum of 36(a-b) (300 MHz, $CDCl_3$):



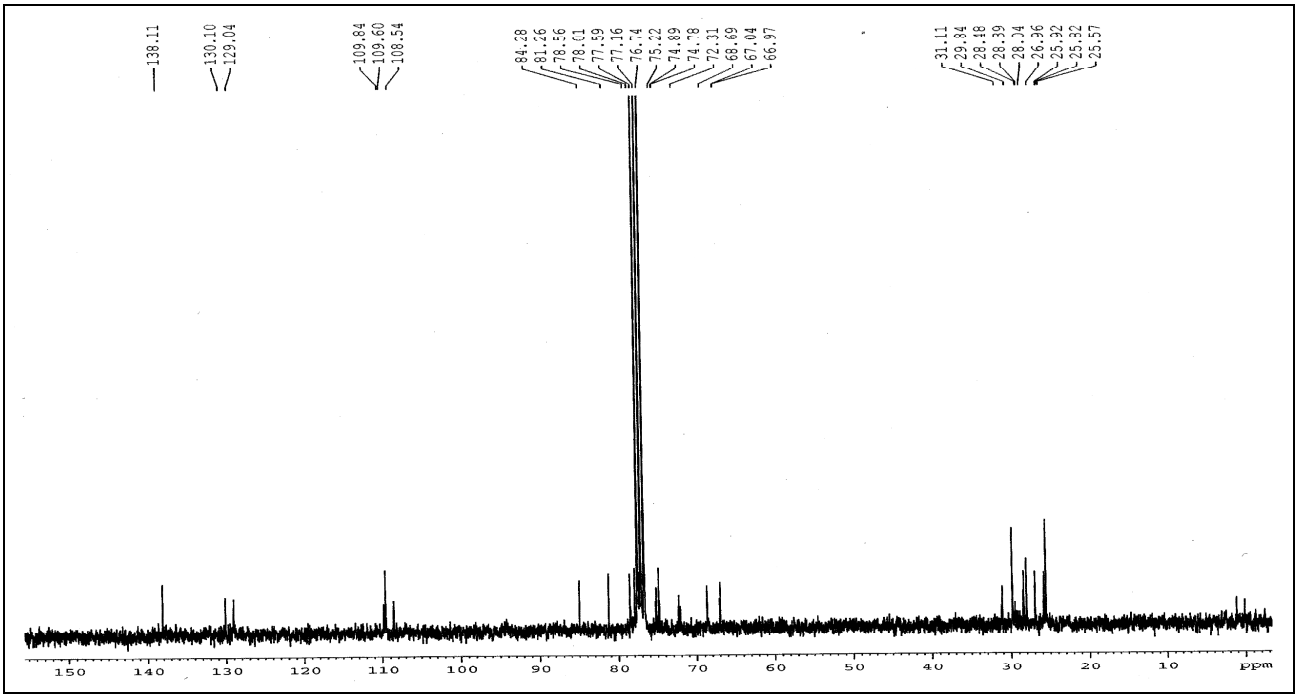
^{13}C NMR spectrum of 36(a-b) (75 MHz, $CDCl_3$):



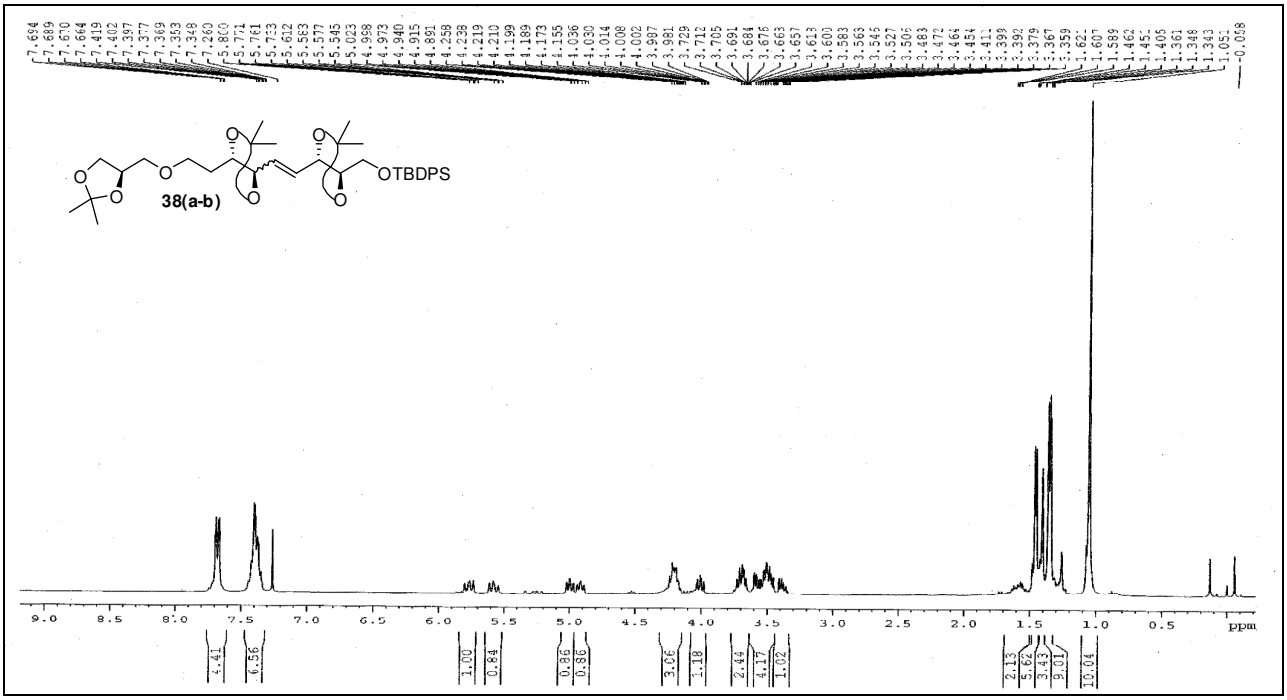
^{1}H NMR spectrum of 37(a-d) (300 MHz, $CDCl_3$):



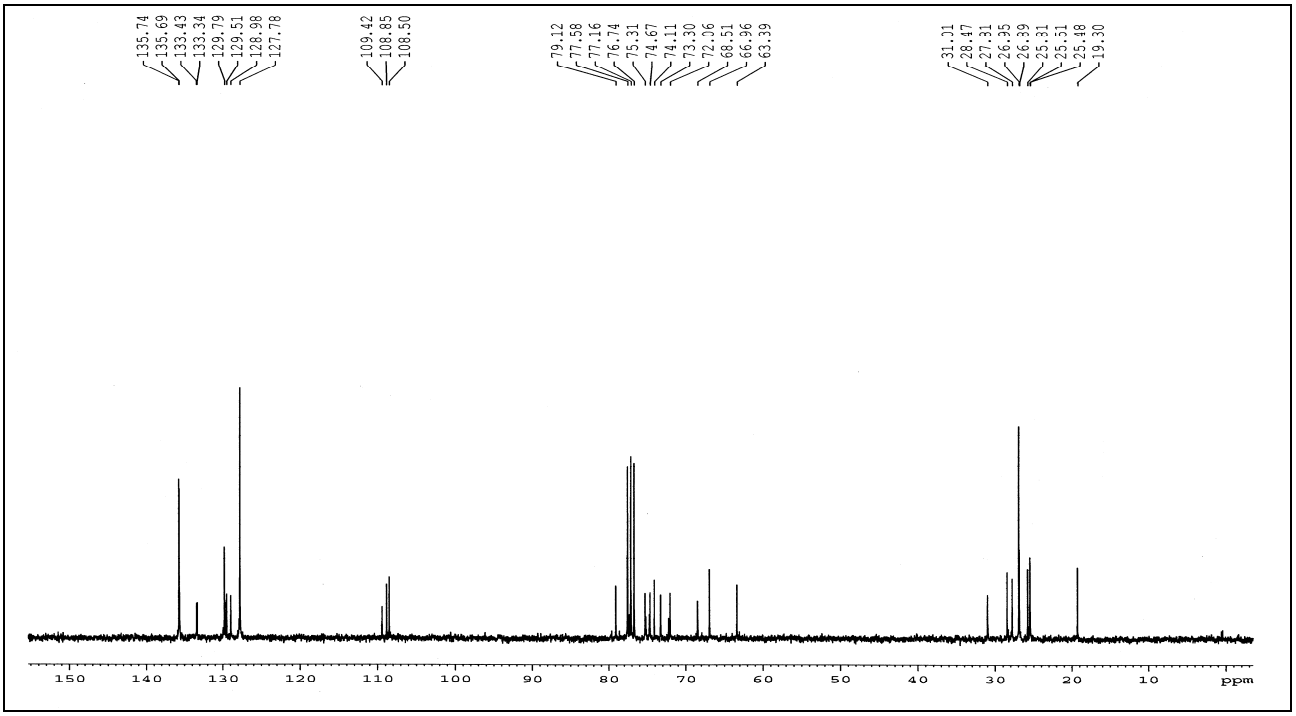
^{13}C NMR spectrum of 37(a-d) (75 MHz, $CDCl_3$):



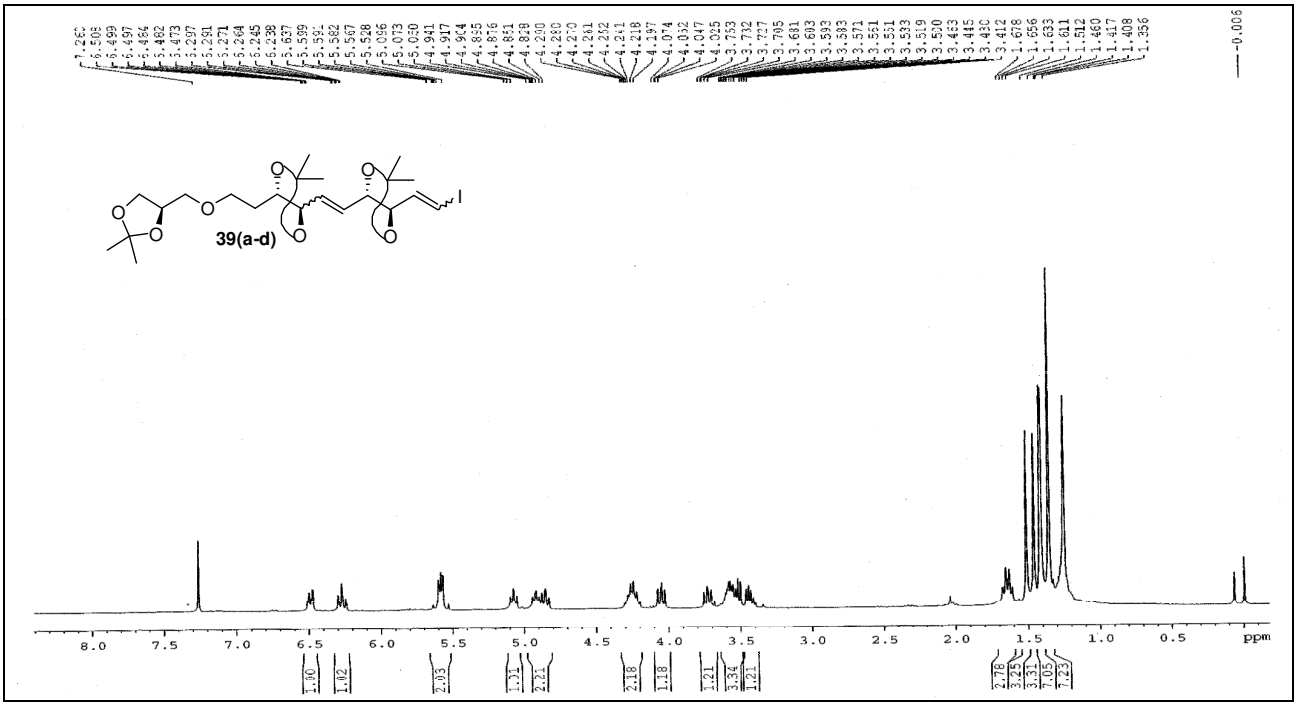
^{1}H NMR spectrum of 38(a-b) (300 MHz, $CDCl_3$):



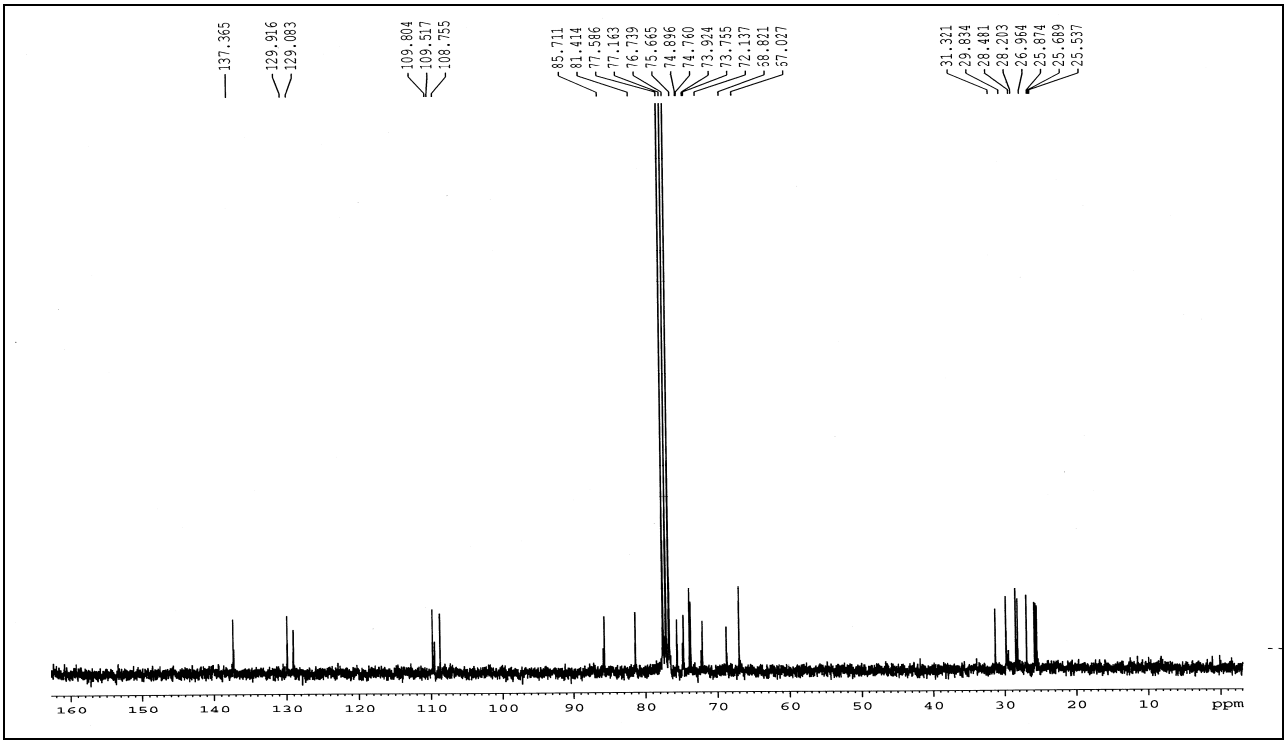
^{13}C NMR spectrum of 38(a-b) (75 MHz, $CDCl_3$):



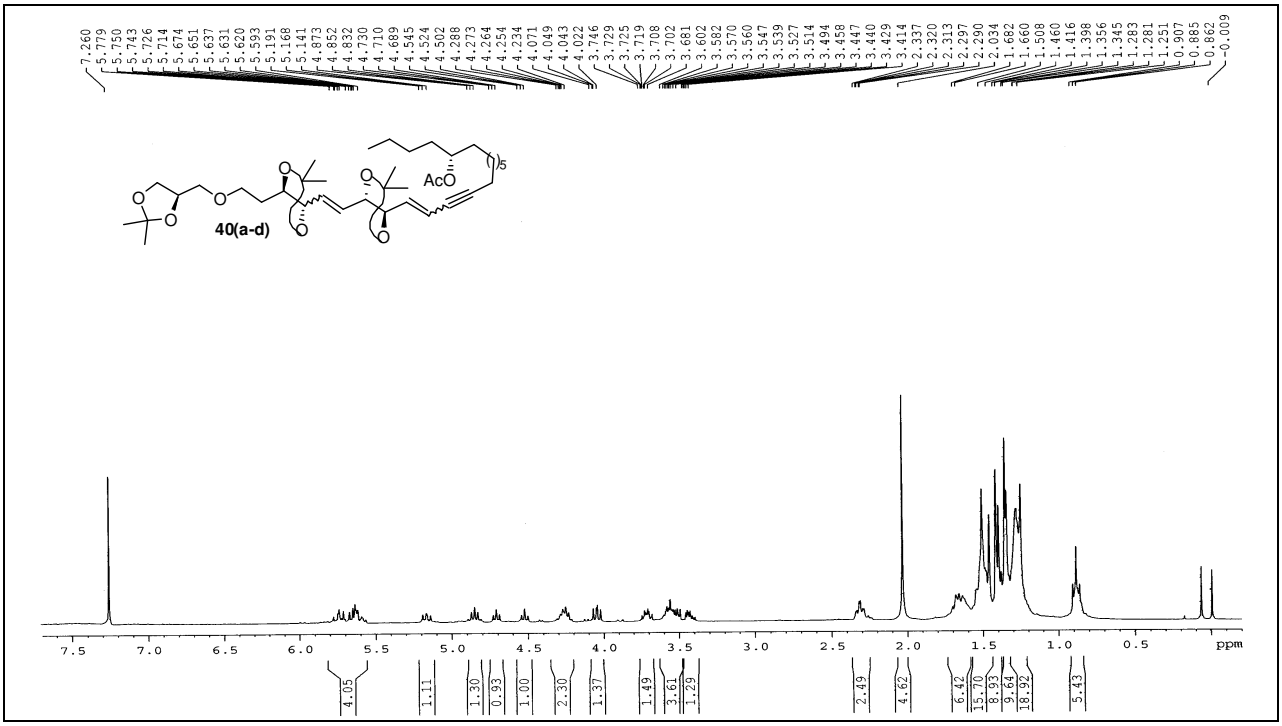
^{1}H NMR spectrum of 39(a-d) (300 MHz, $CDCl_3$):



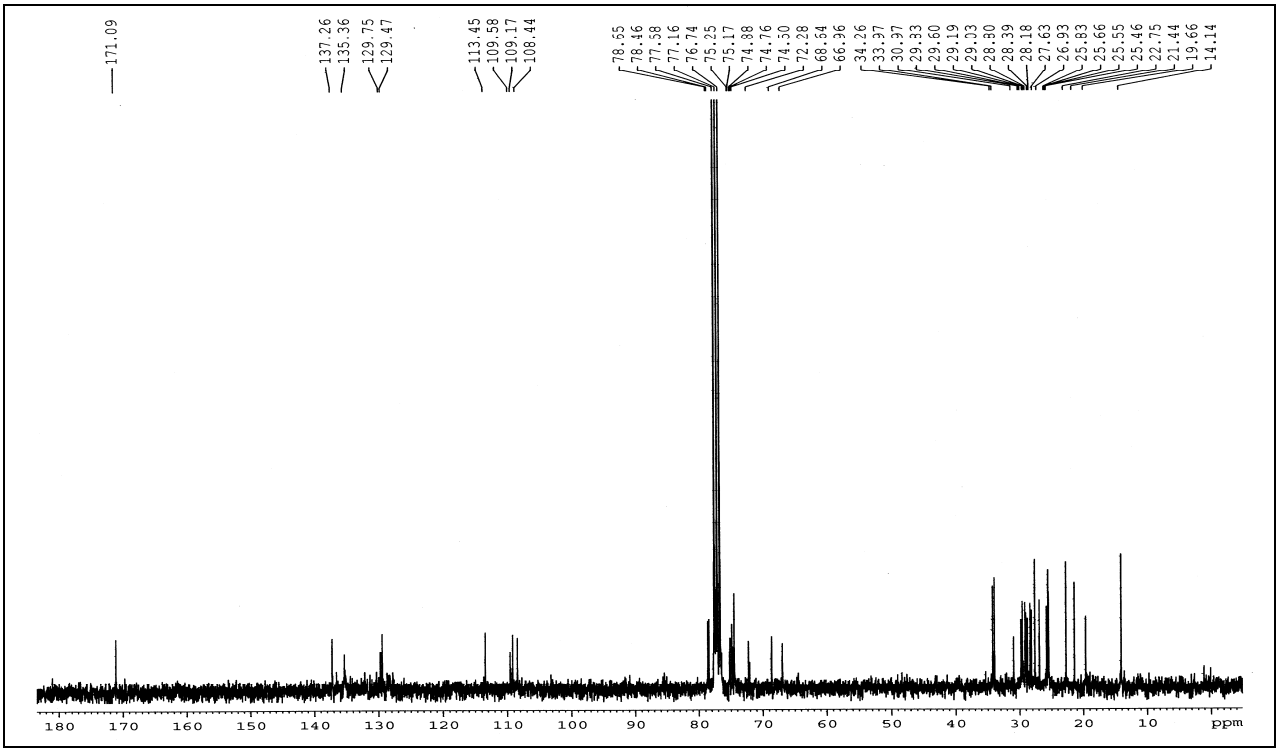
^{13}C NMR spectrum of 39(a-d) (75 MHz, $CDCl_3$):



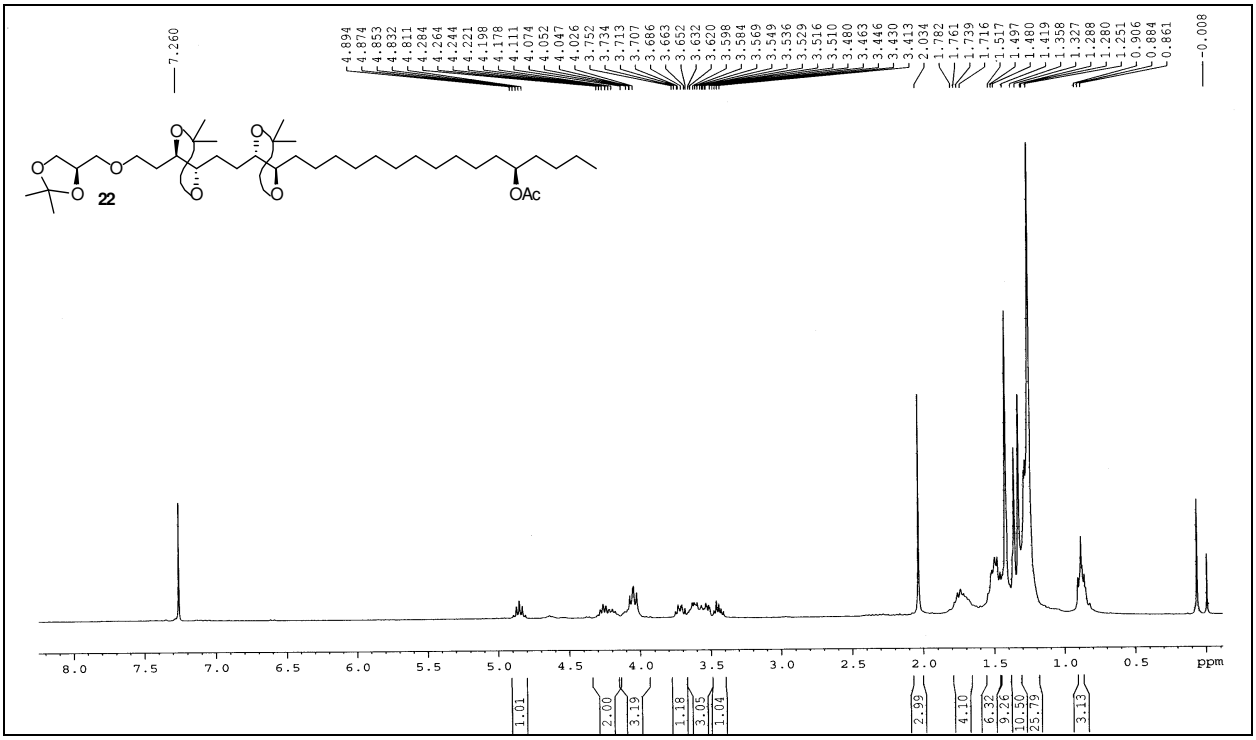
^{1}H NMR spectrum of 40 (a-d) (300 MHz, $CDCl_3$):



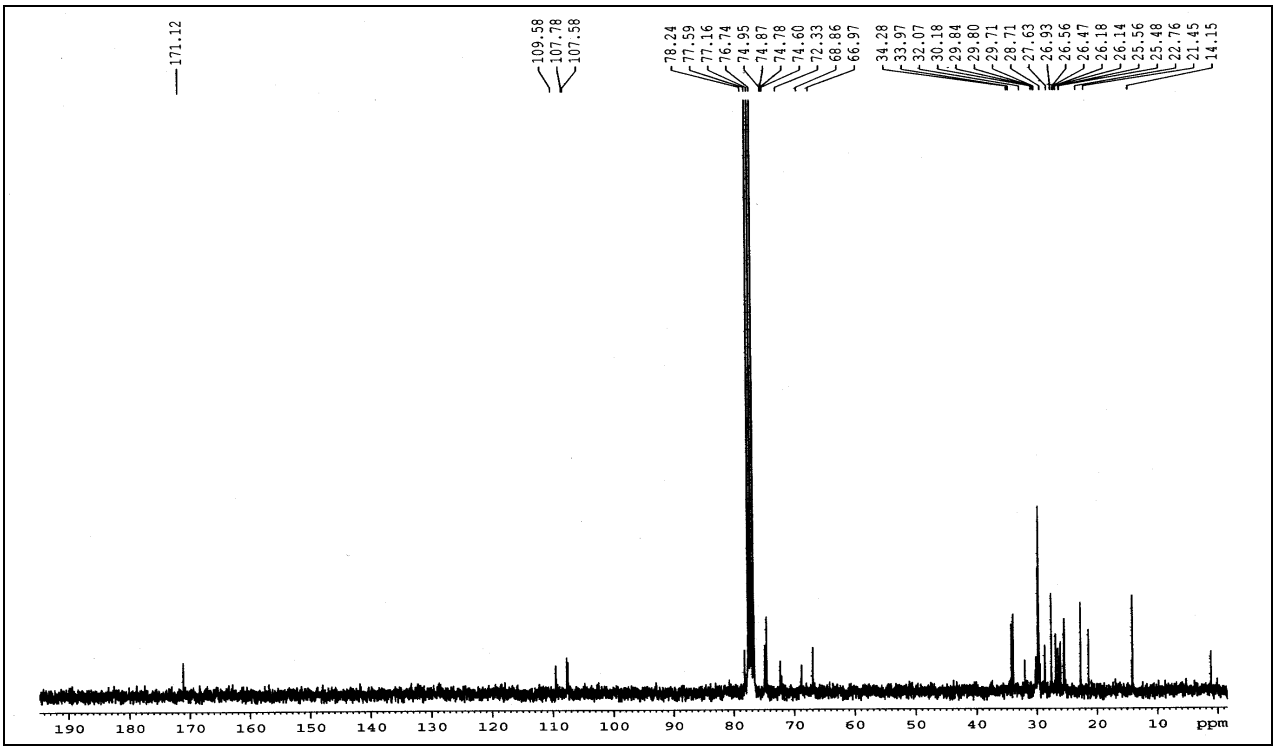
^{13}C NMR spectrum of 40 (a-d) (75 MHz, $CDCl_3$):



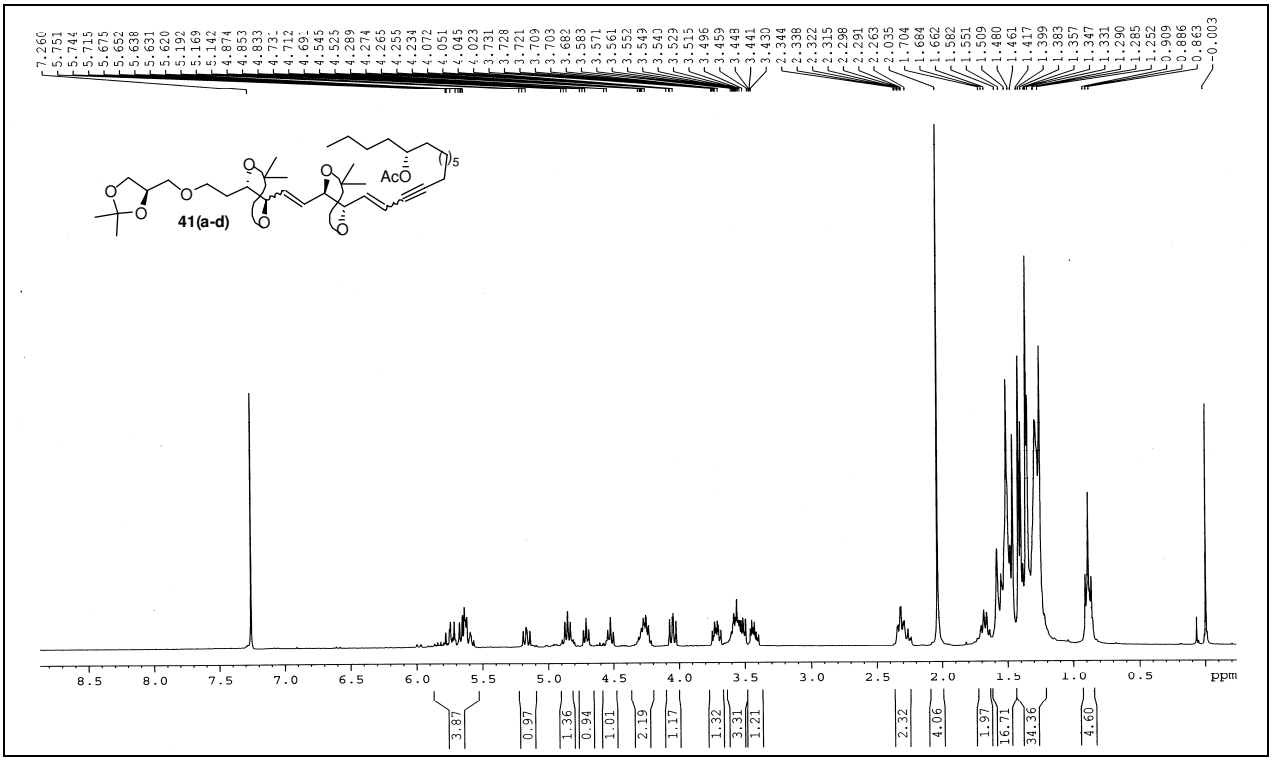
^{1}H NMR spectrum of 22 (300 MHz, $CDCl_3$):



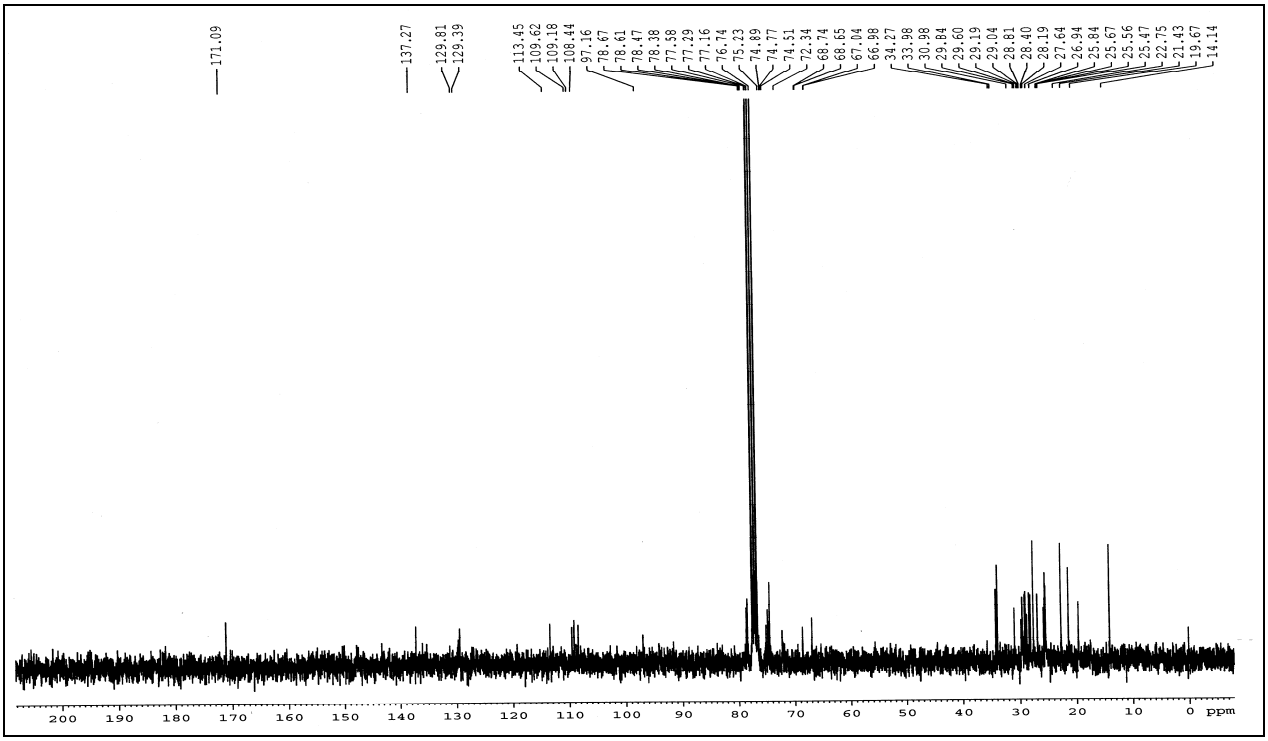
^{13}C NMR spectrum of 22 (75 MHz, $CDCl_3$):



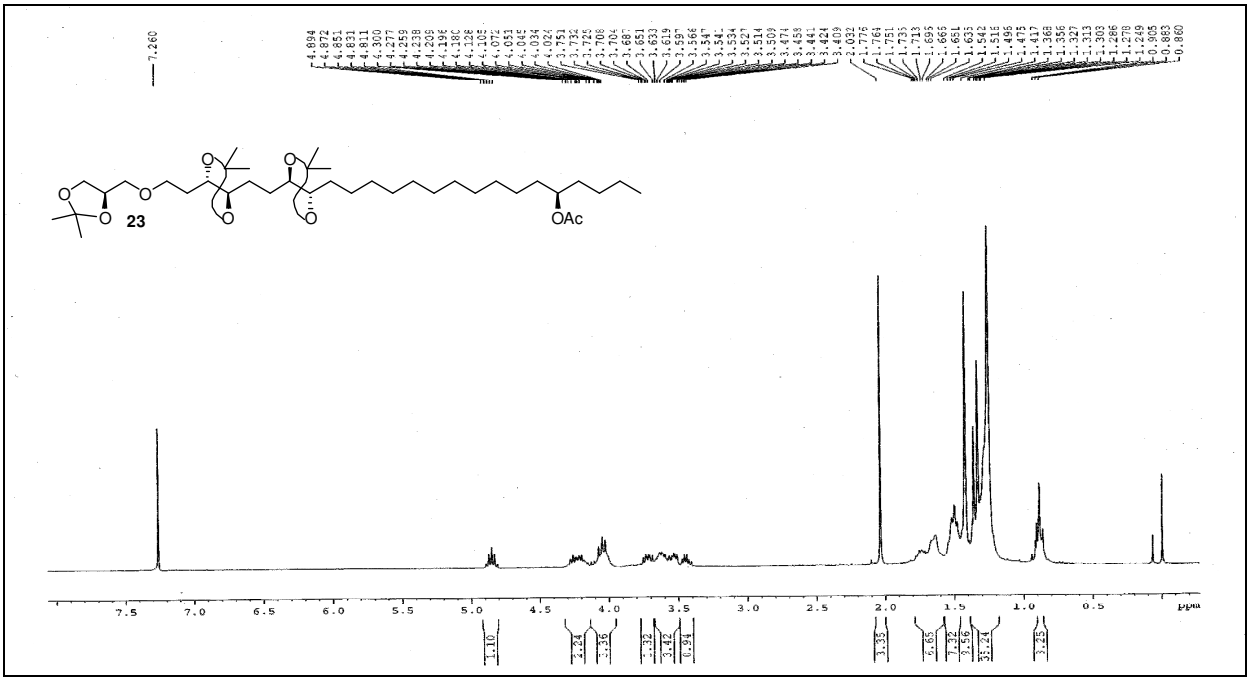
^{1}H NMR spectrum of 41 (a-d) (300 MHz, $CDCl_3$):



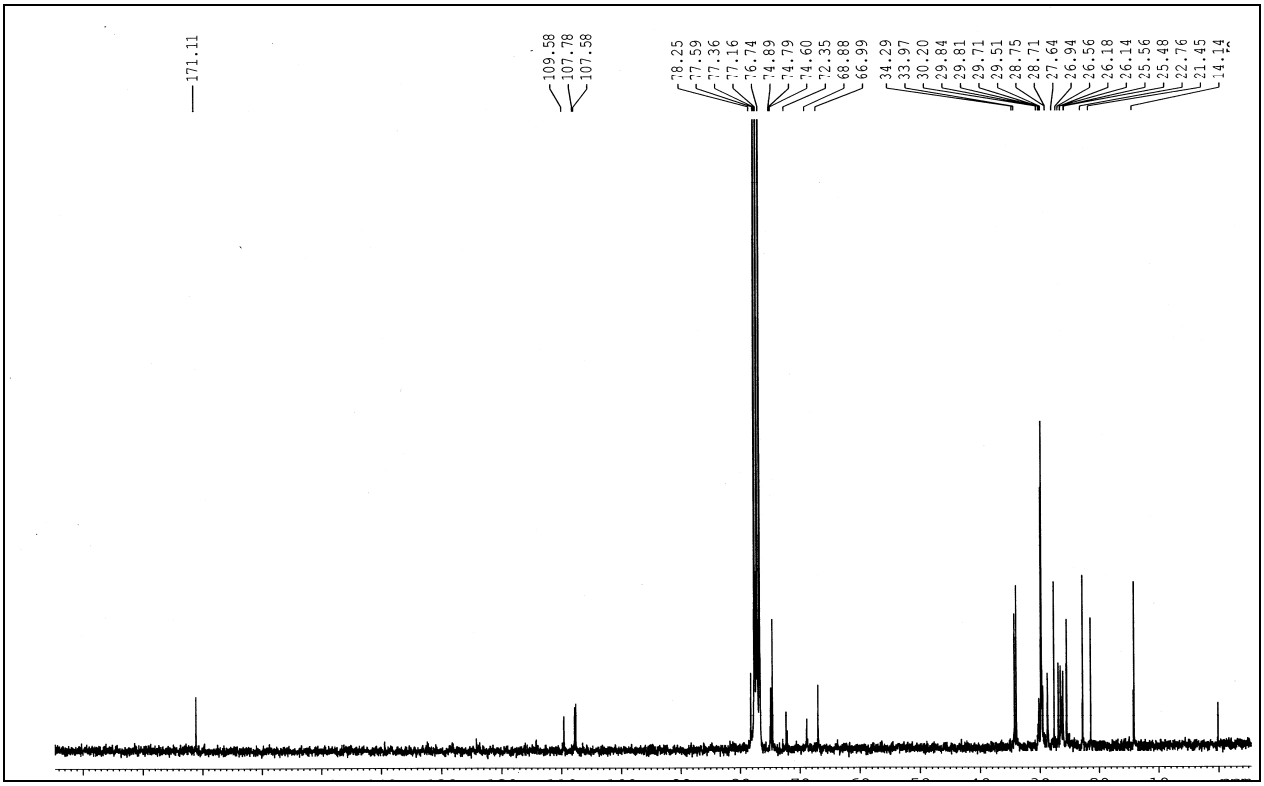
^{13}C NMR spectrum of 41 (a-d) (75 MHz, $CDCl_3$):



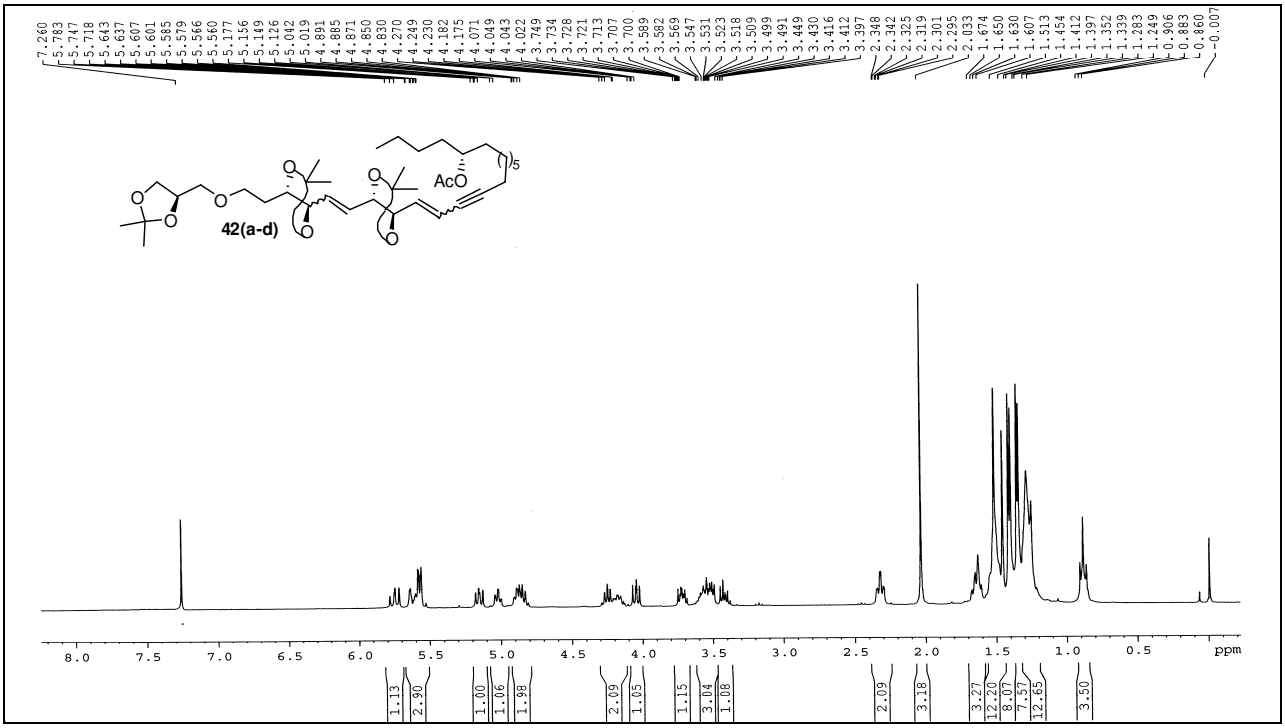
^{1}H NMR spectrum of 23 (300 MHz, $CDCl_3$):



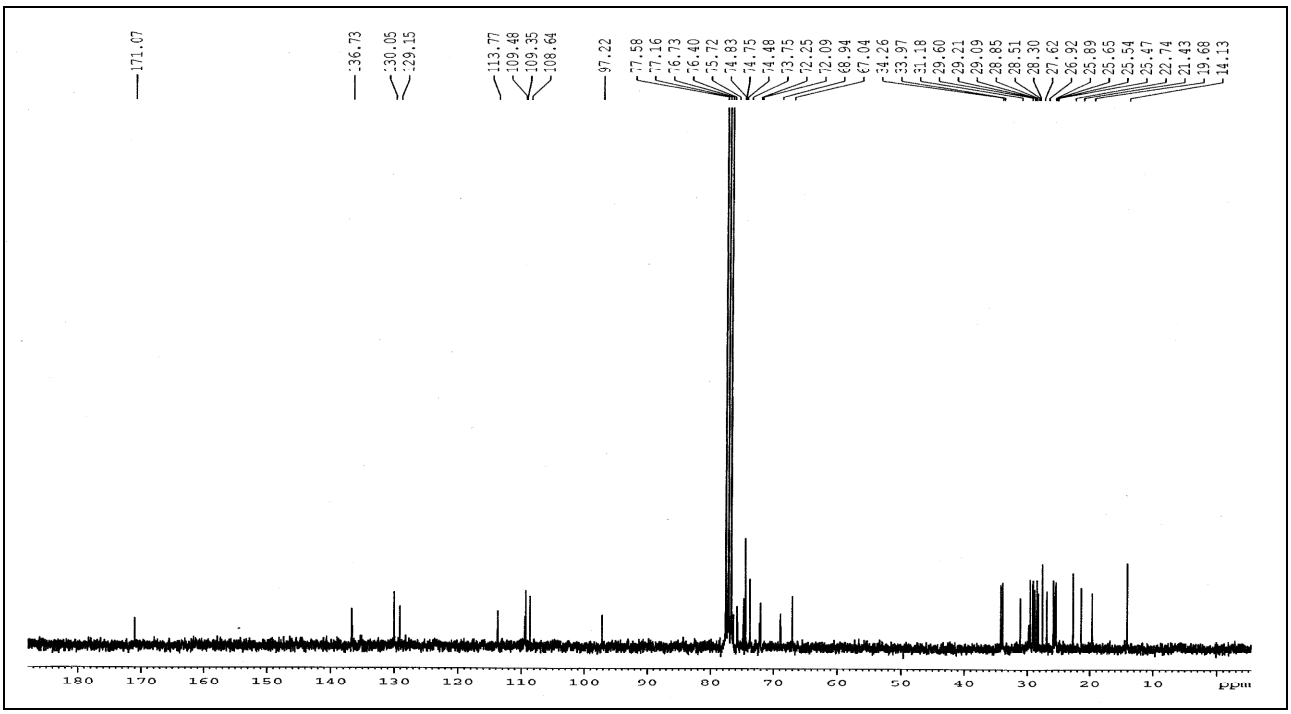
^{13}C NMR spectrum of 23 (75 MHz, $CDCl_3$):



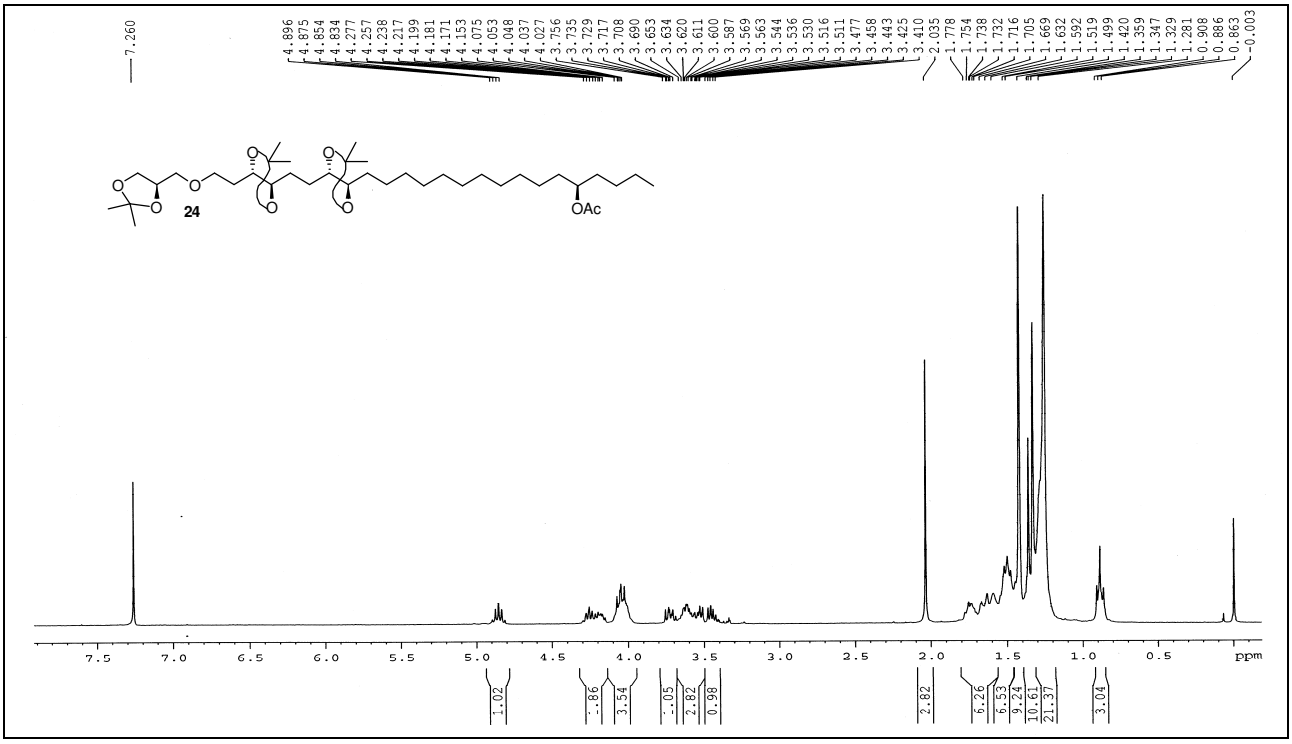
^{1}H NMR spectrum of 42(a-d) (300 MHz, $CDCl_3$):



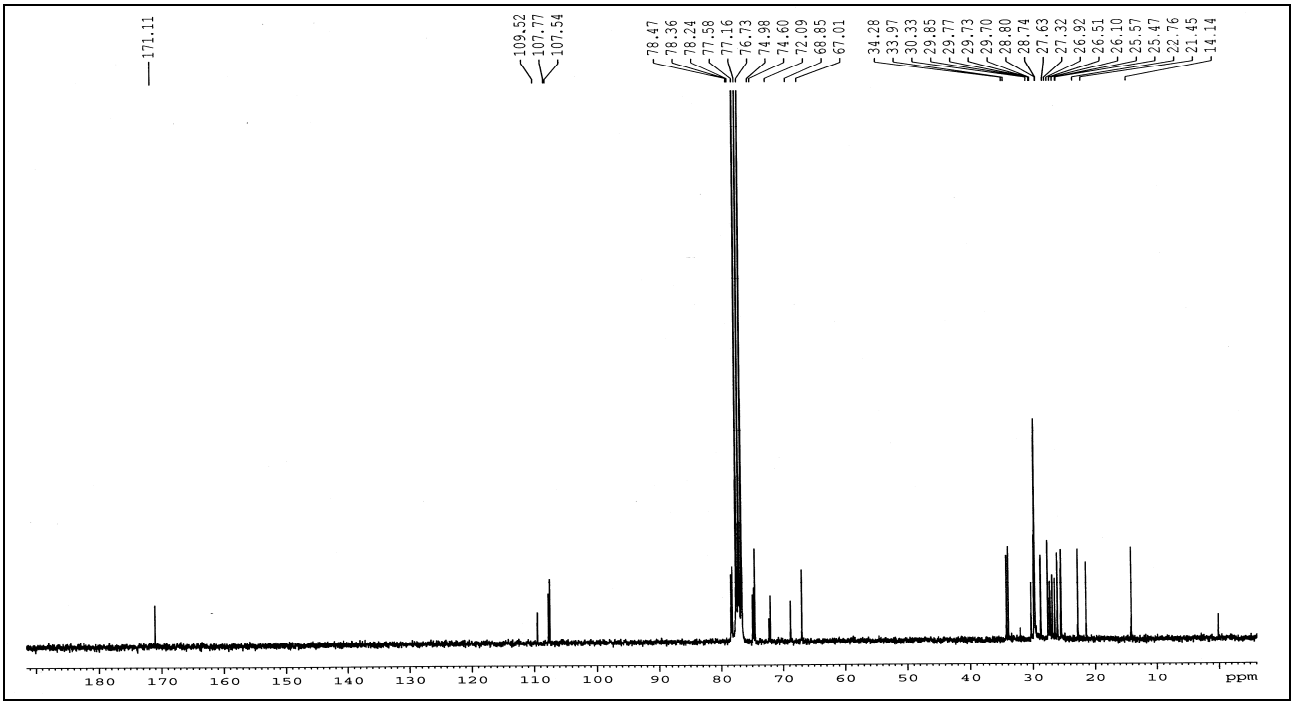
^{13}C NMR spectrum of 42(a-d) (75 MHz, $CDCl_3$):



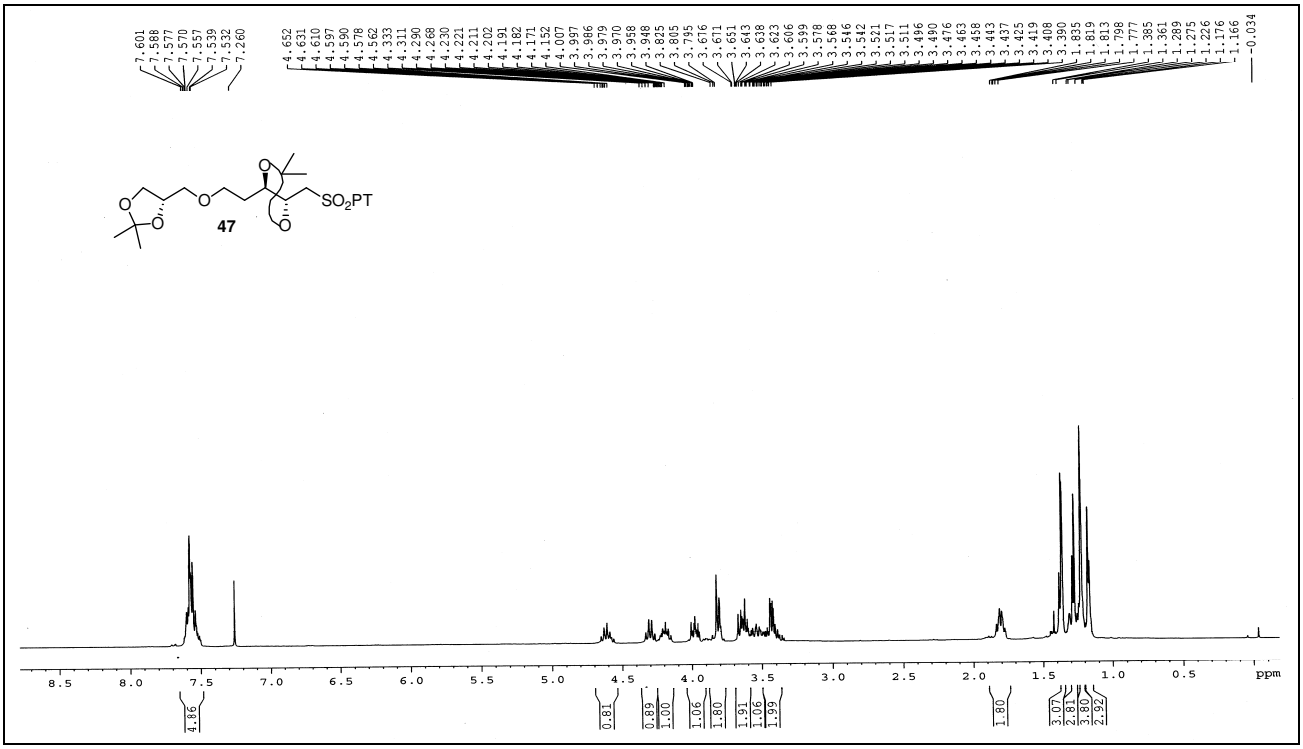
^{1}H NMR spectrum of 24 (300 MHz, $CDCl_3$):



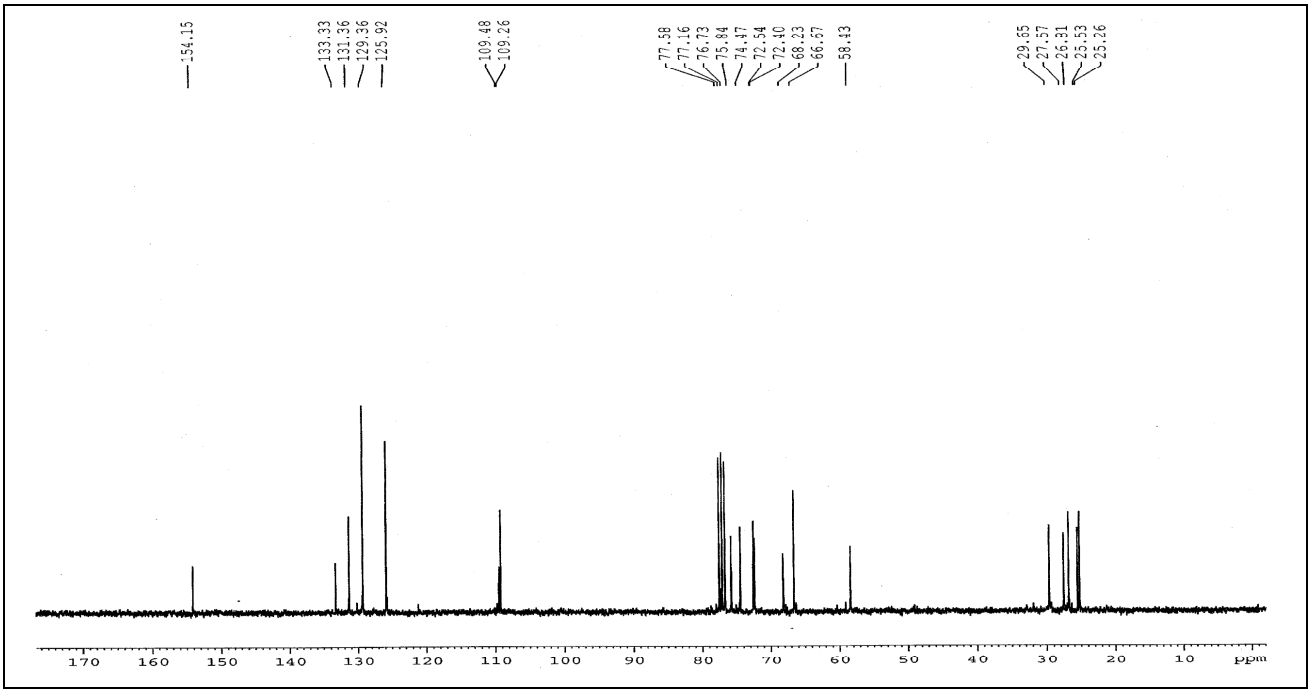
^{13}C NMR spectrum of 24 (75 MHz, $CDCl_3$):



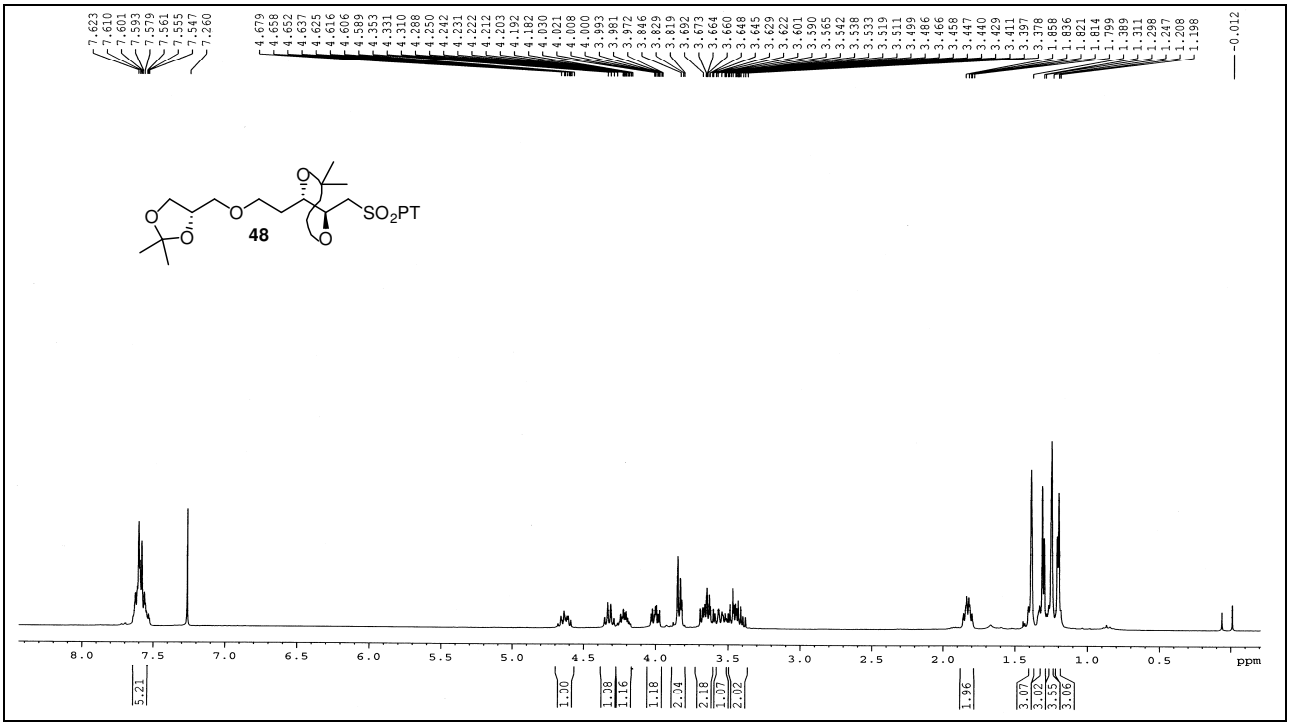
^{1}H NMR spectrum of 47 (300 MHz, $CDCl_3$):



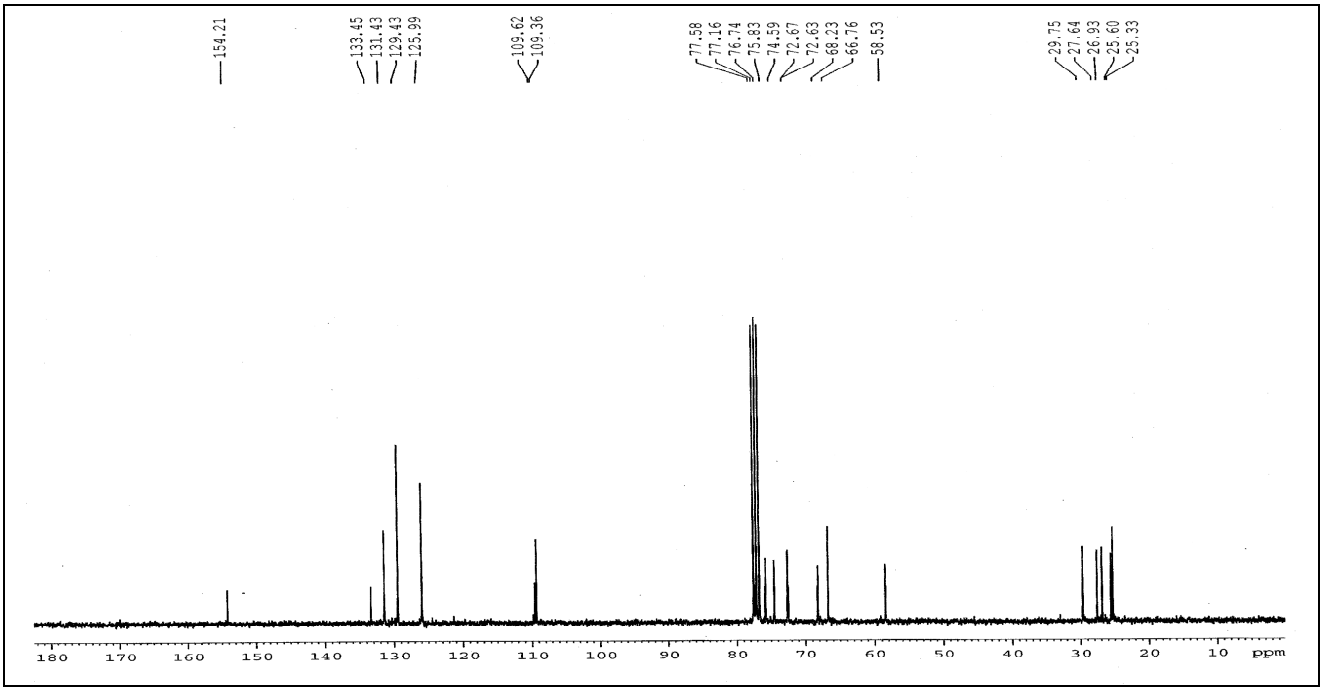
^{13}C NMR spectrum of 47 (75 MHz, $CDCl_3$):



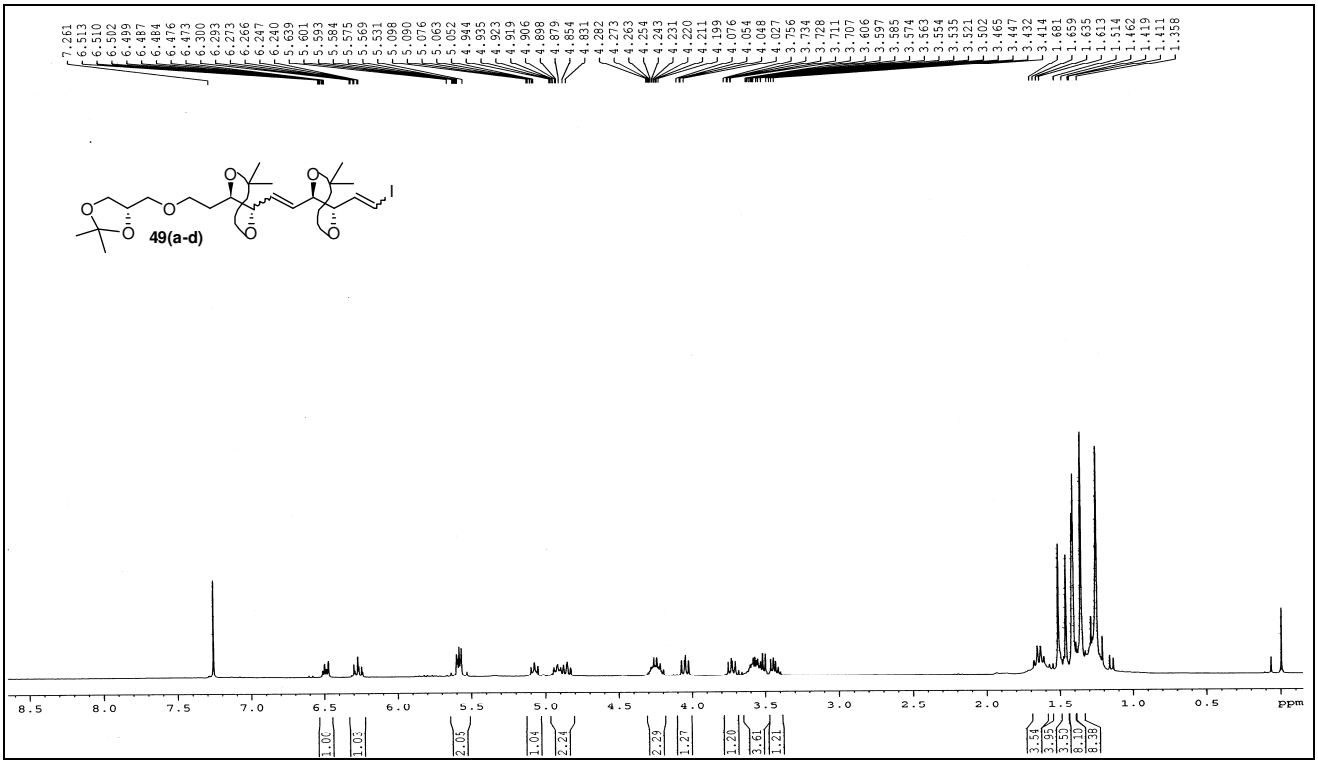
^{1}H NMR spectrum of 48 (300 MHz, $CDCl_3$):



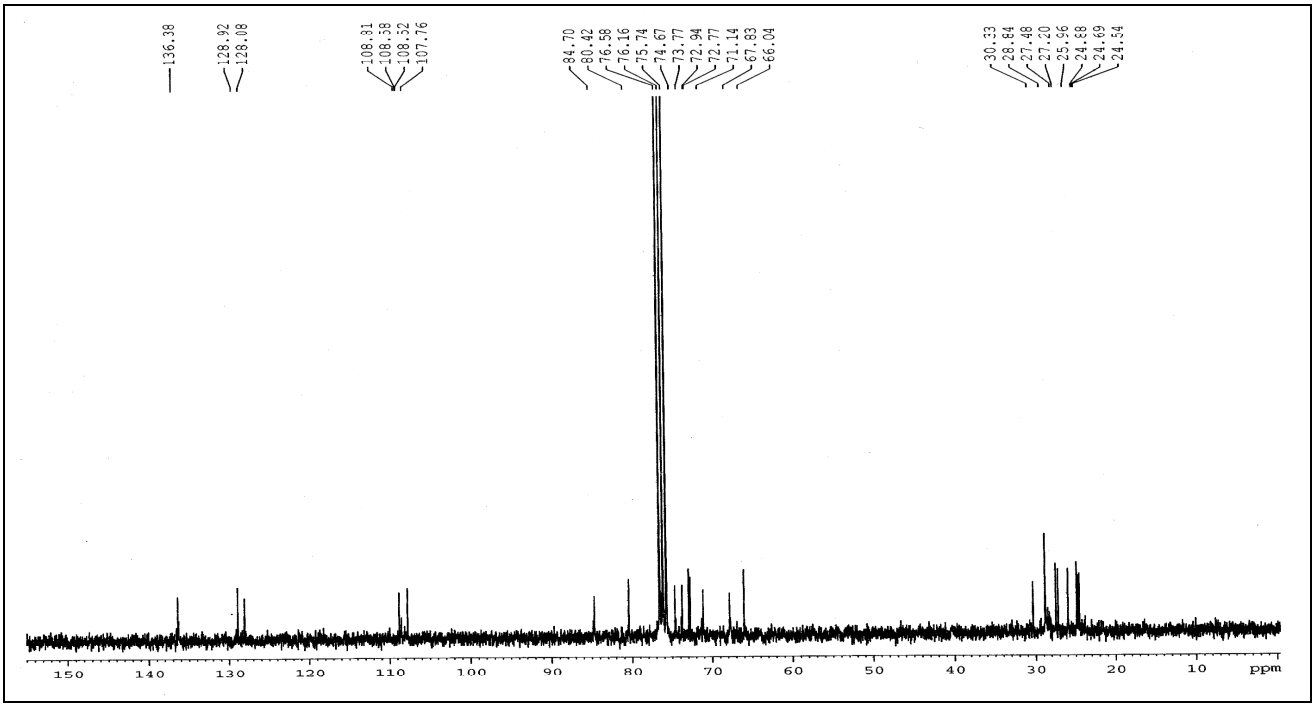
^{13}C NMR spectrum of 48 (75 MHz, $CDCl_3$):



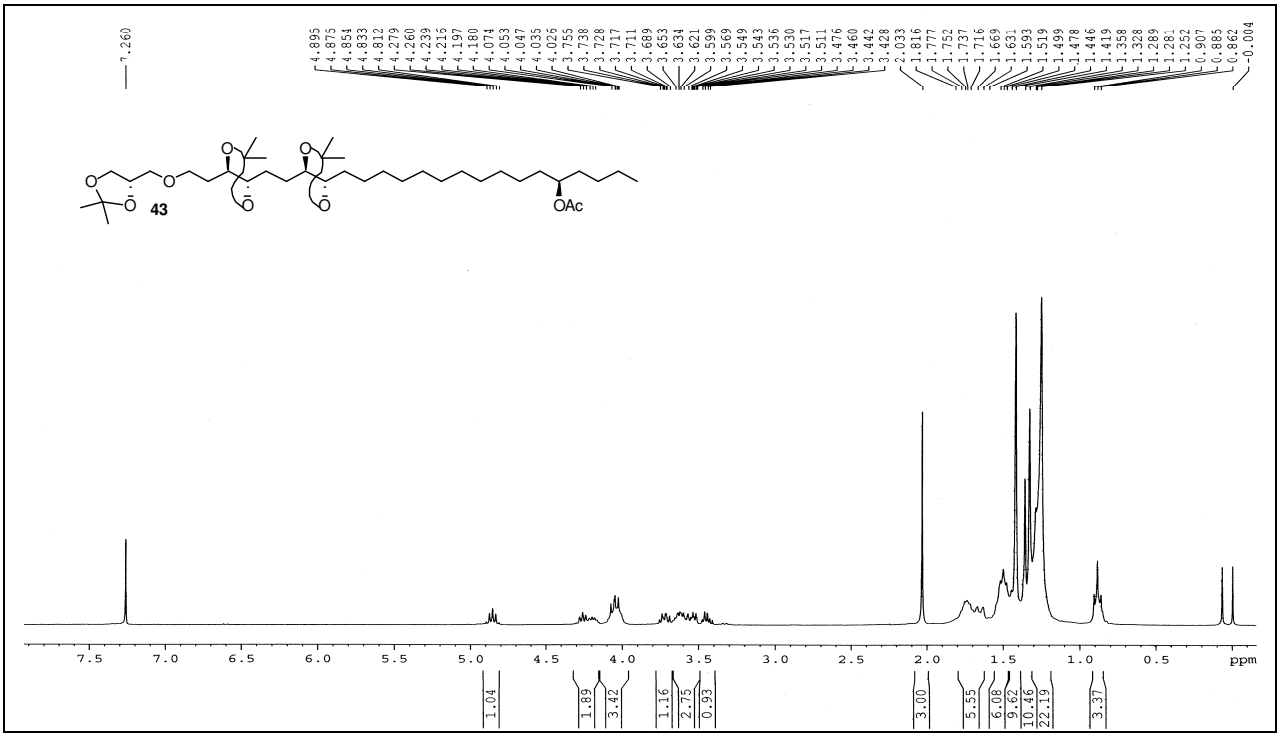
^{1}H NMR spectrum of 49(a-d) (300 MHz, $CDCl_3$):



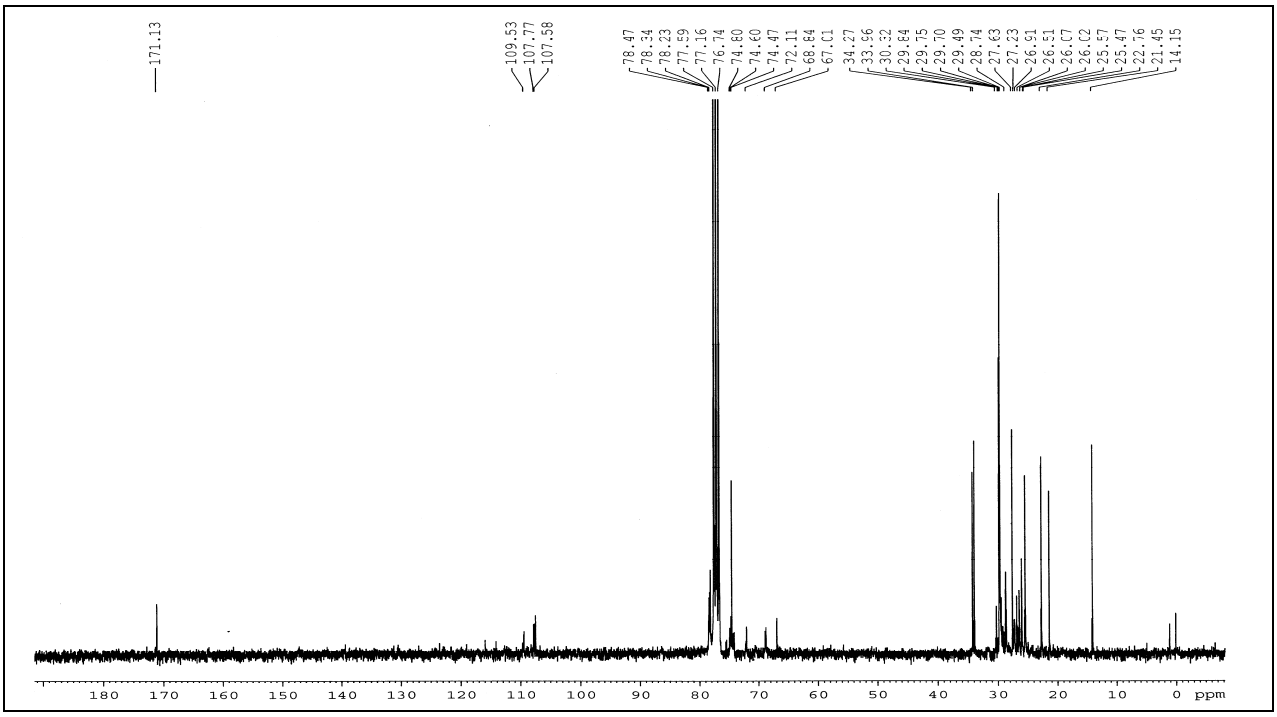
^{13}C NMR spectrum of 49 (a-d) (75 MHz, $CDCl_3$):



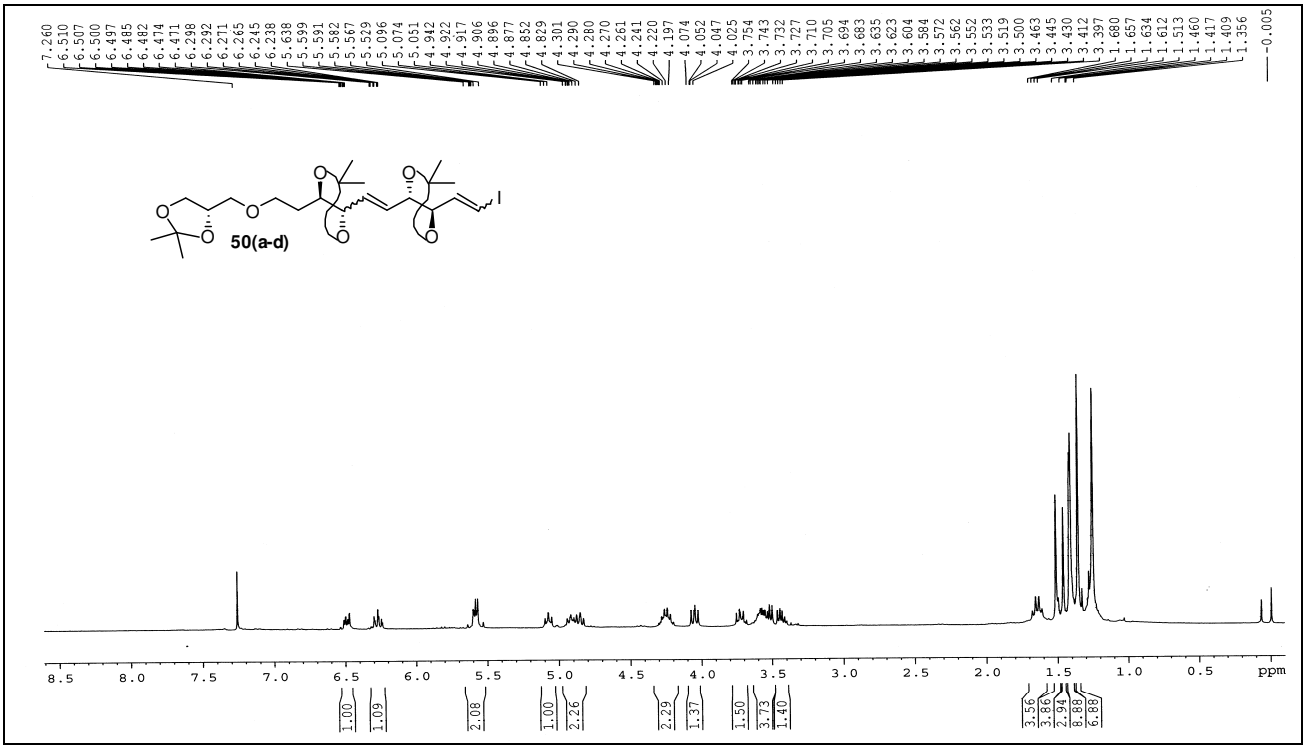
^{1}H NMR spectrum of 43 (300 MHz, $CDCl_3$):



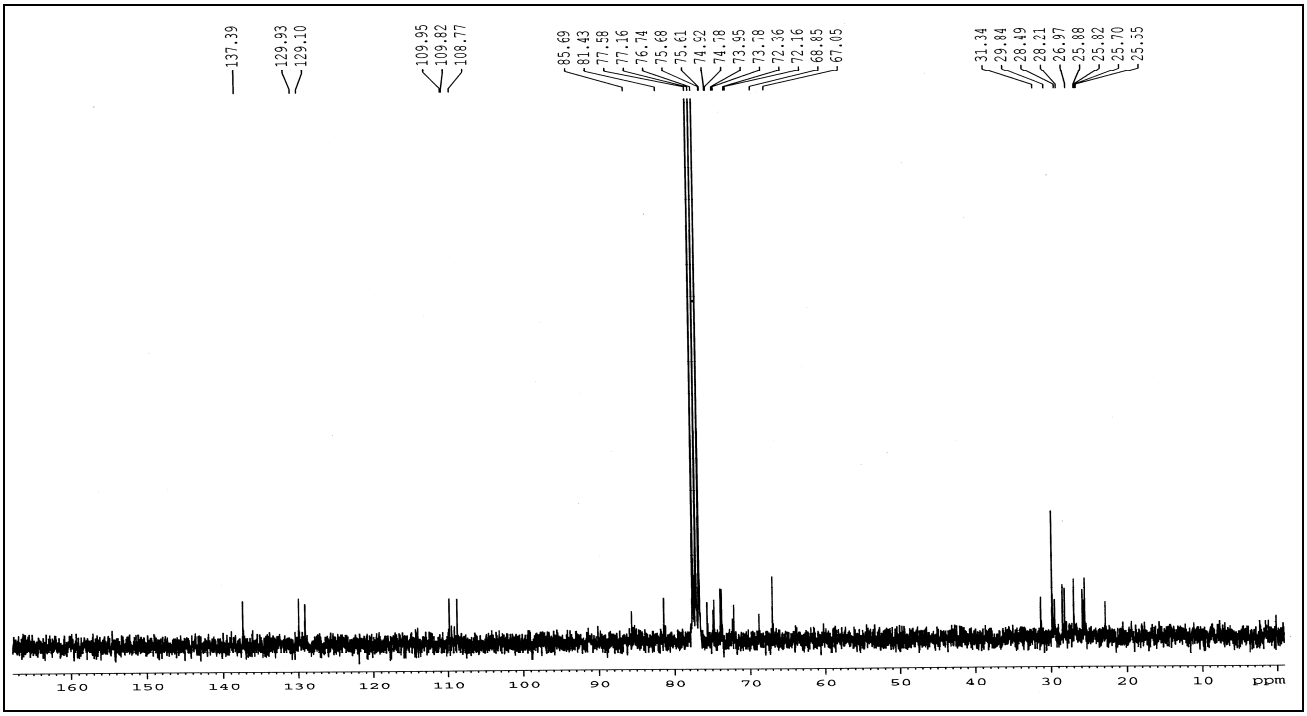
^{13}C NMR spectrum of 43 (75 MHz, $CDCl_3$):



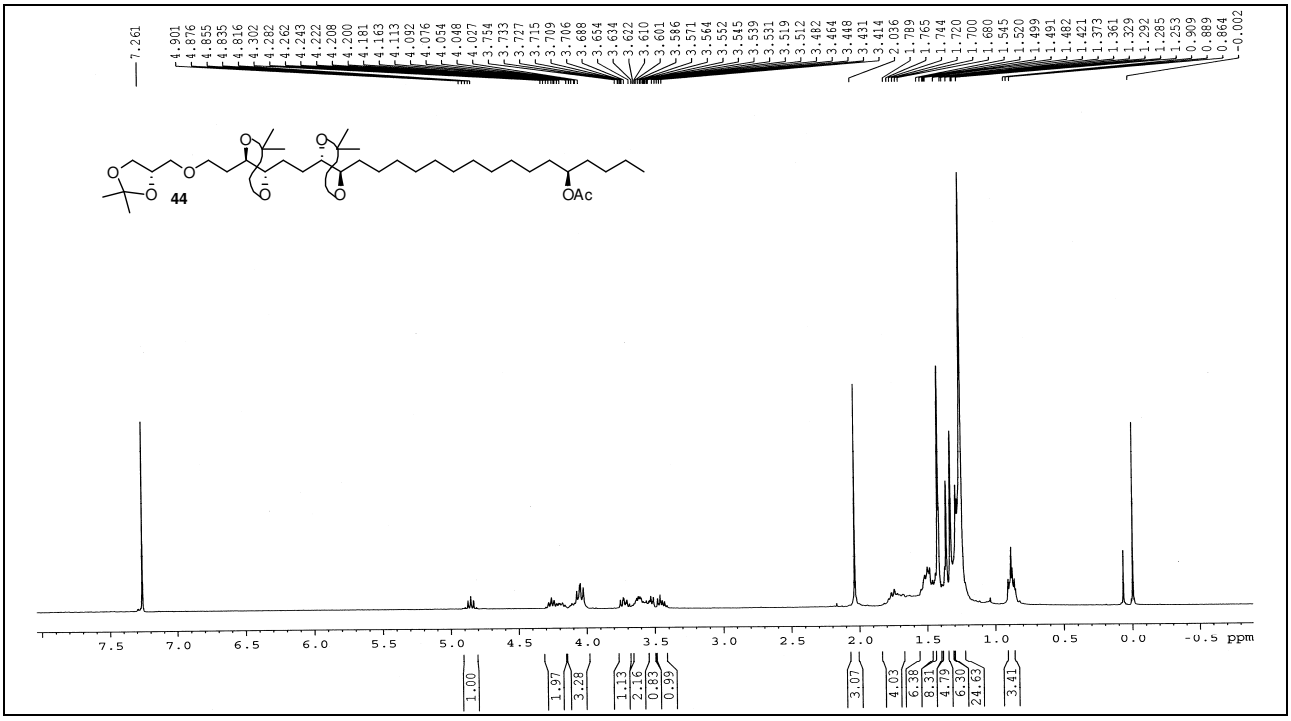
^{1}H NMR spectrum of 50(a-d) (300 MHz, $CDCl_3$):



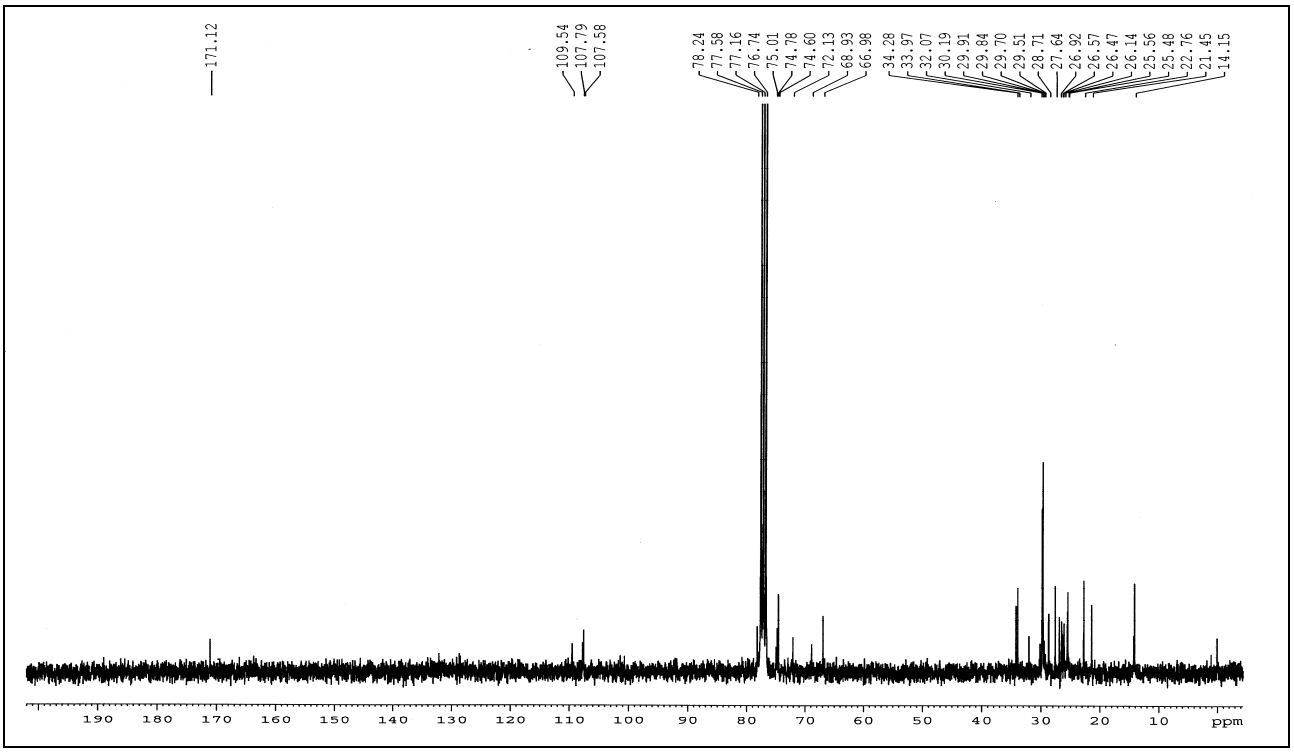
^{13}C NMR spectrum of 50 (a-d) (75 MHz, $CDCl_3$):



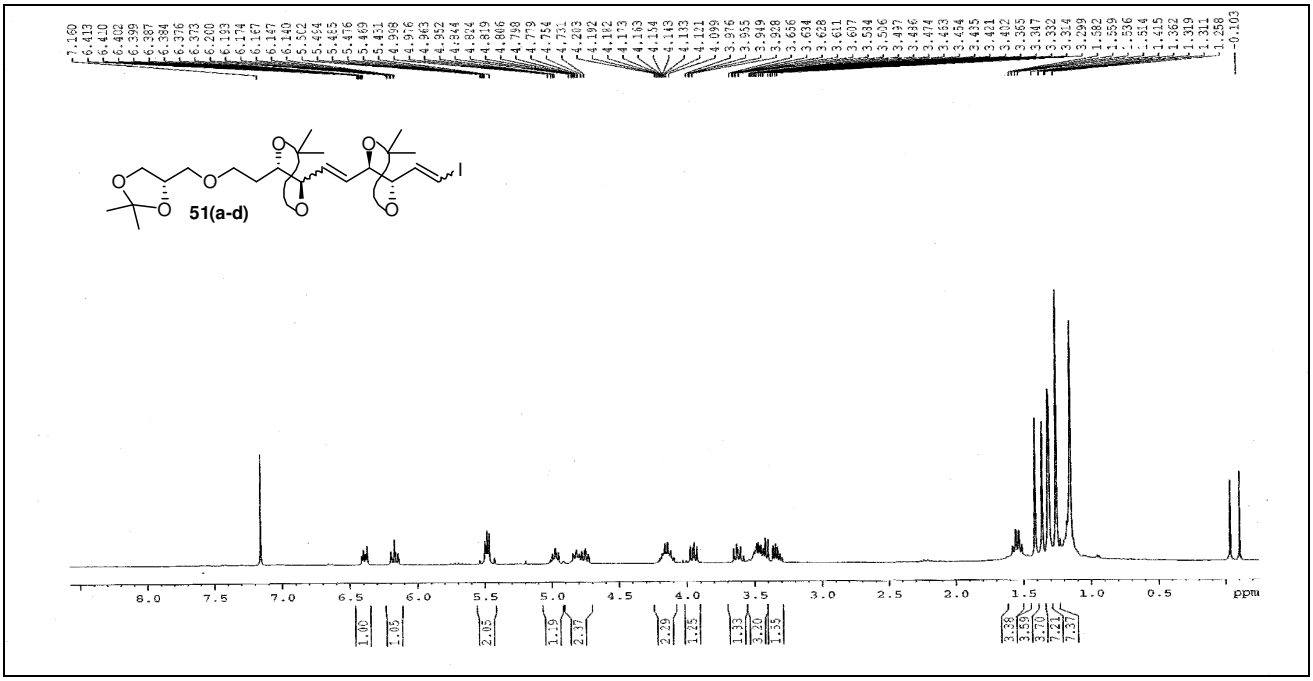
^{1}H NMR spectrum of 44 (300 MHz, $CDCl_3$):



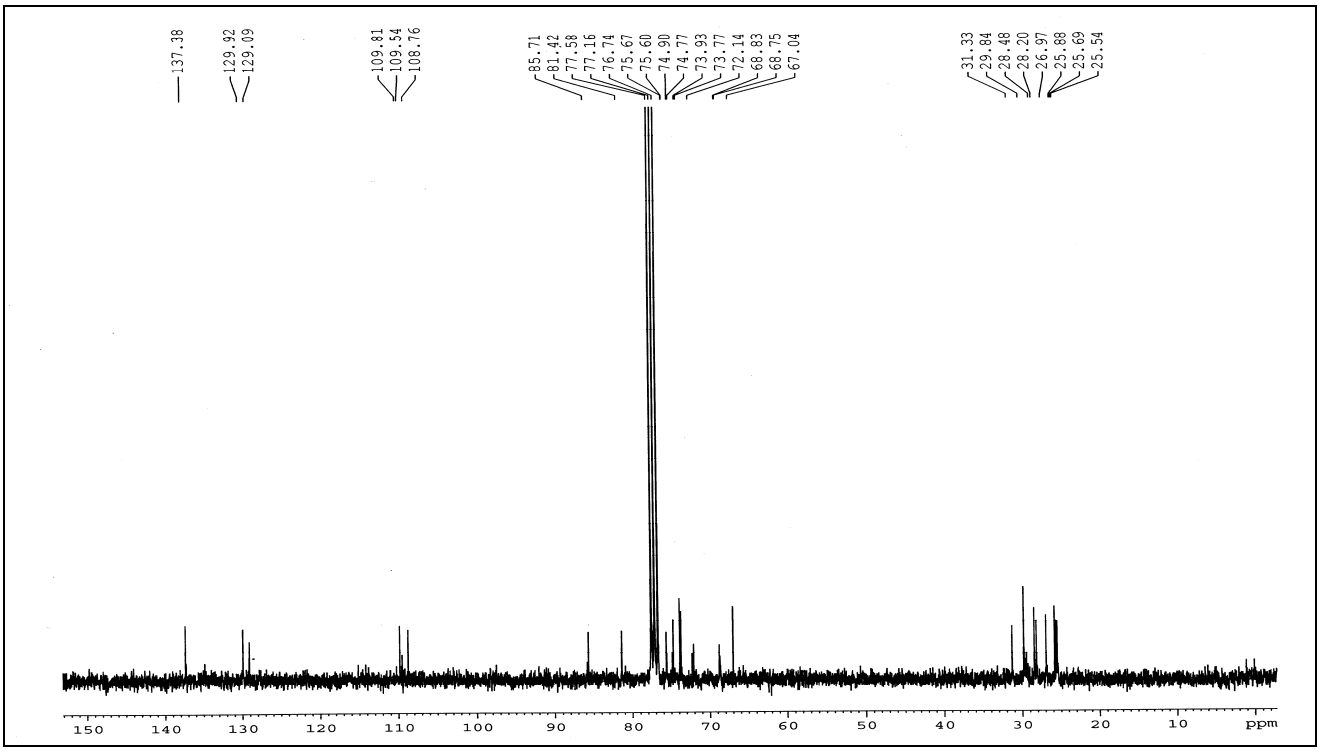
^{13}C NMR spectrum of 44 (75 MHz, $CDCl_3$):



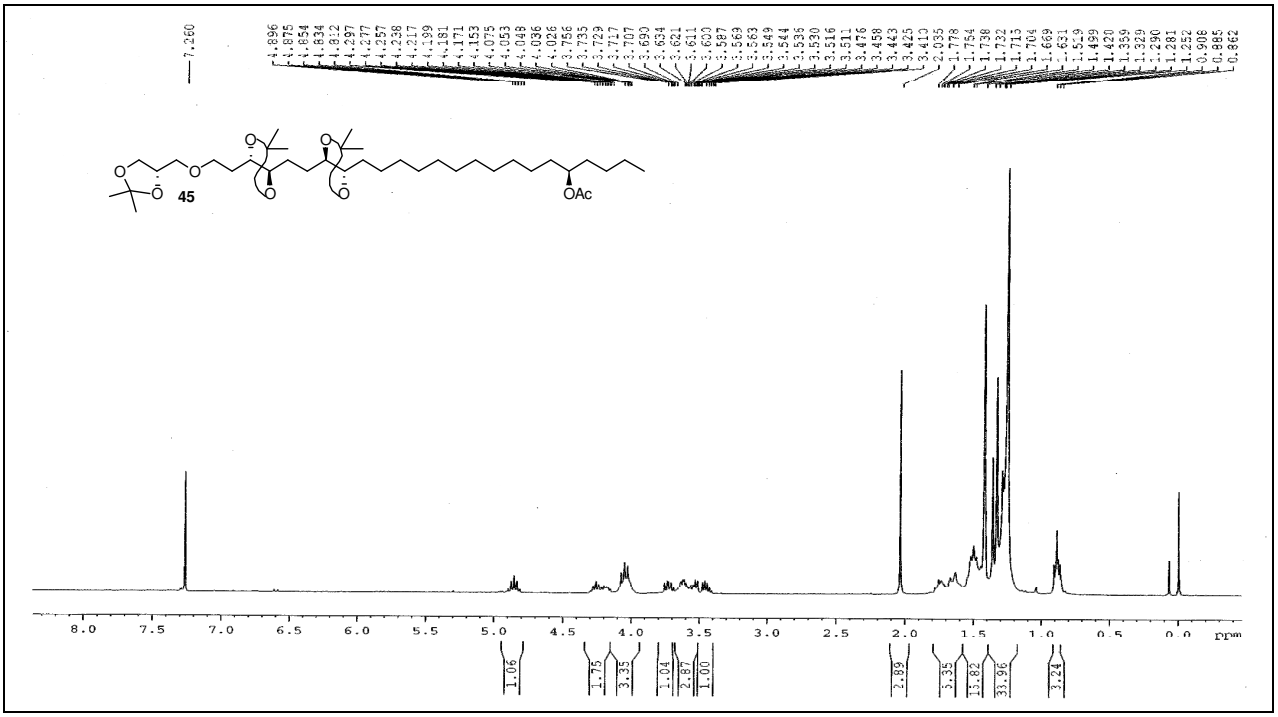
^{1}H NMR spectrum of 51 (a-d) (300 MHz, $CDCl_3$):



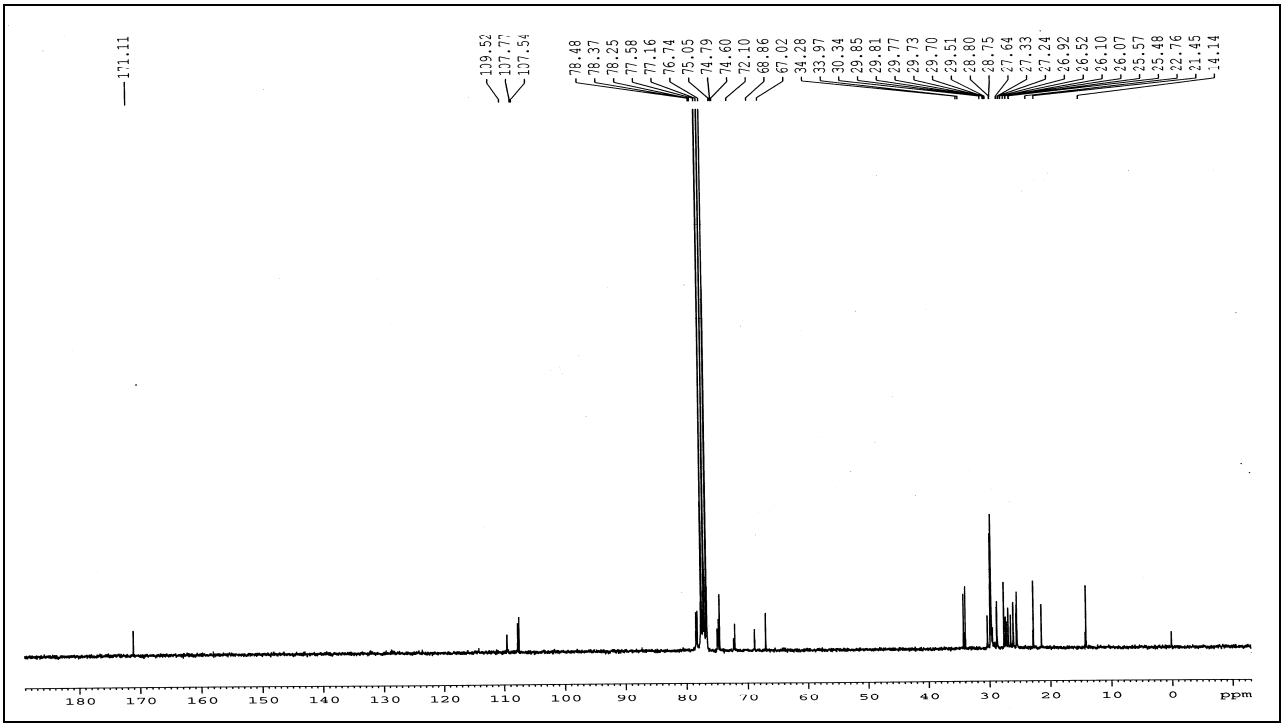
^{13}C NMR spectrum of 51 (a-d) (75 MHz, $CDCl_3$):



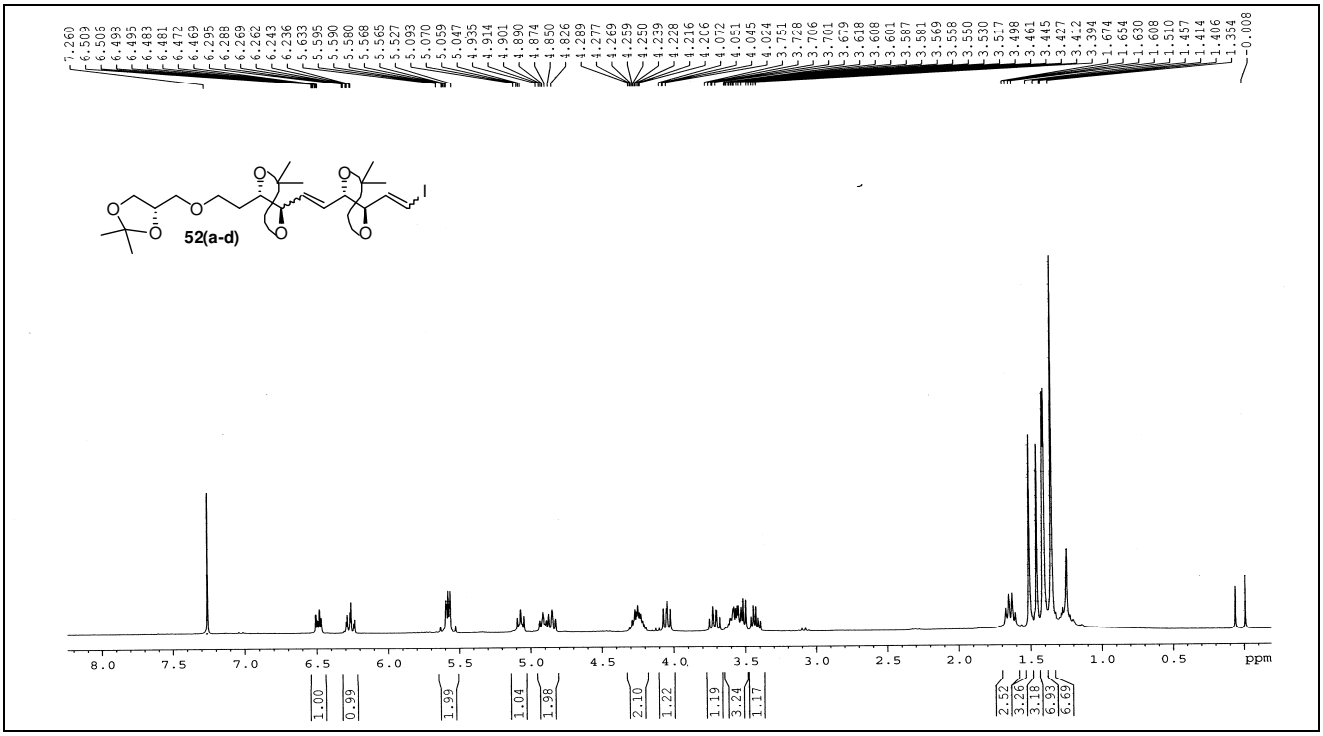
^{1}H NMR spectrum of 45 (300 MHz, $CDCl_3$):



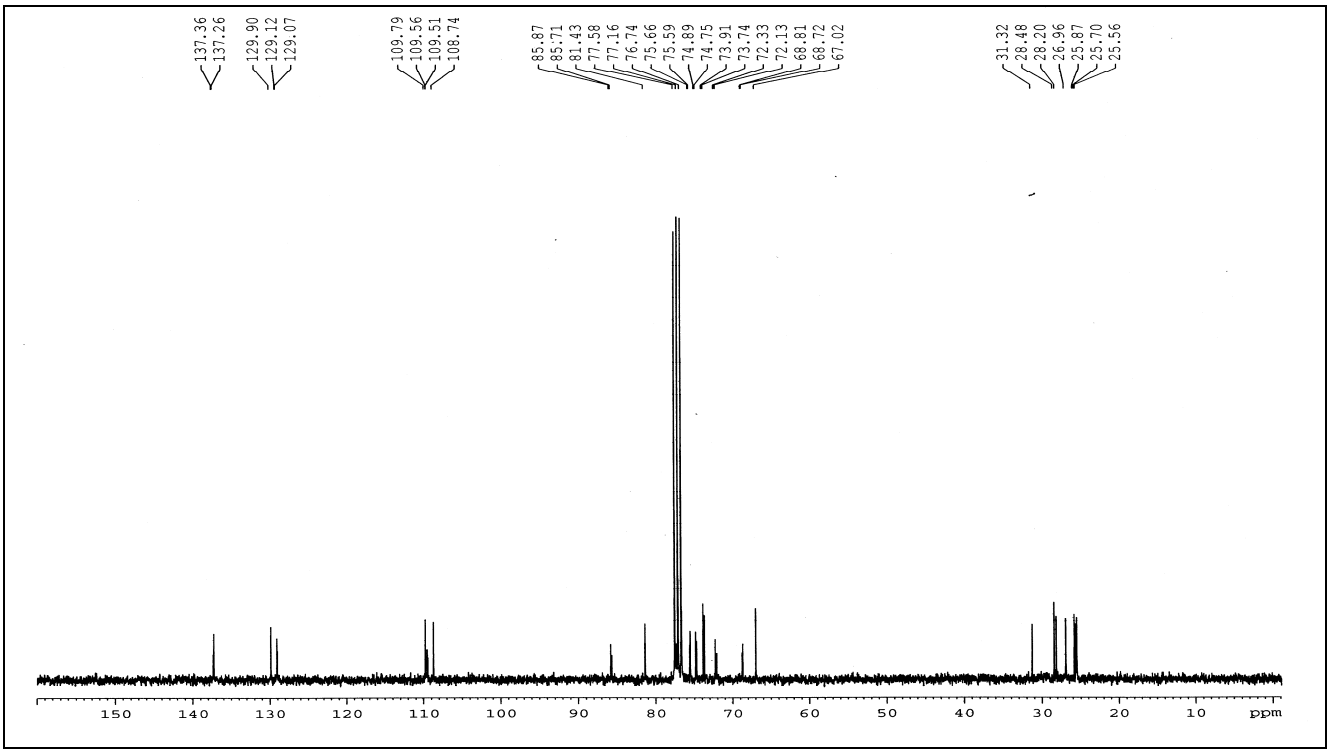
^{13}C NMR spectrum of 45 (75 MHz, $CDCl_3$):



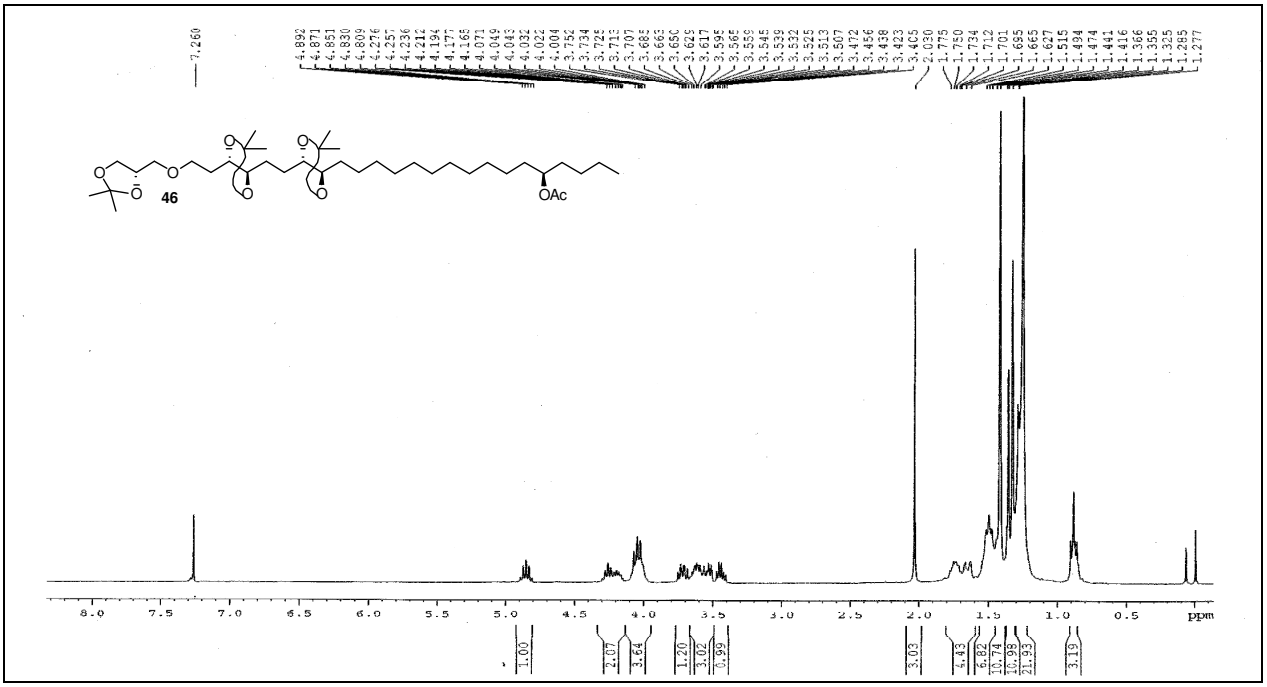
^{1}H NMR spectrum of 52 (a-d) (300 MHz, $CDCl_3$):



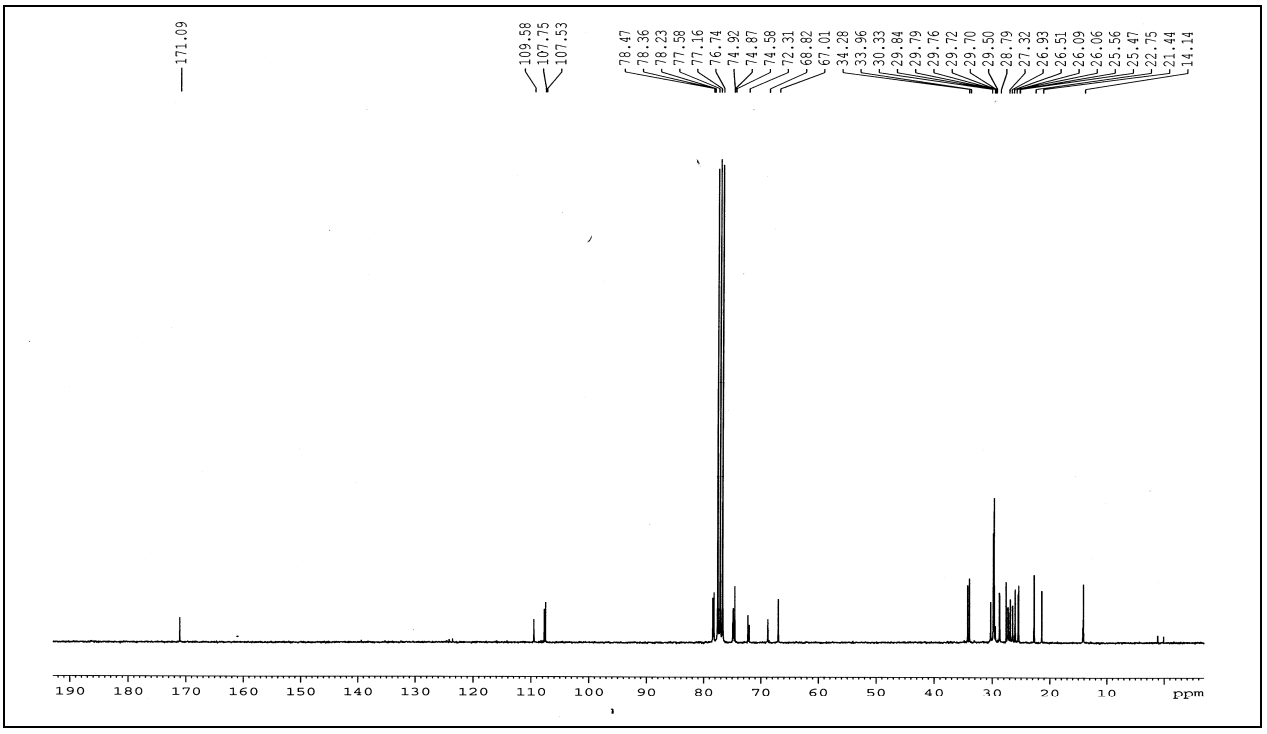
^{13}C NMR spectrum of 52 (a-d) (75 MHz, $CDCl_3$):



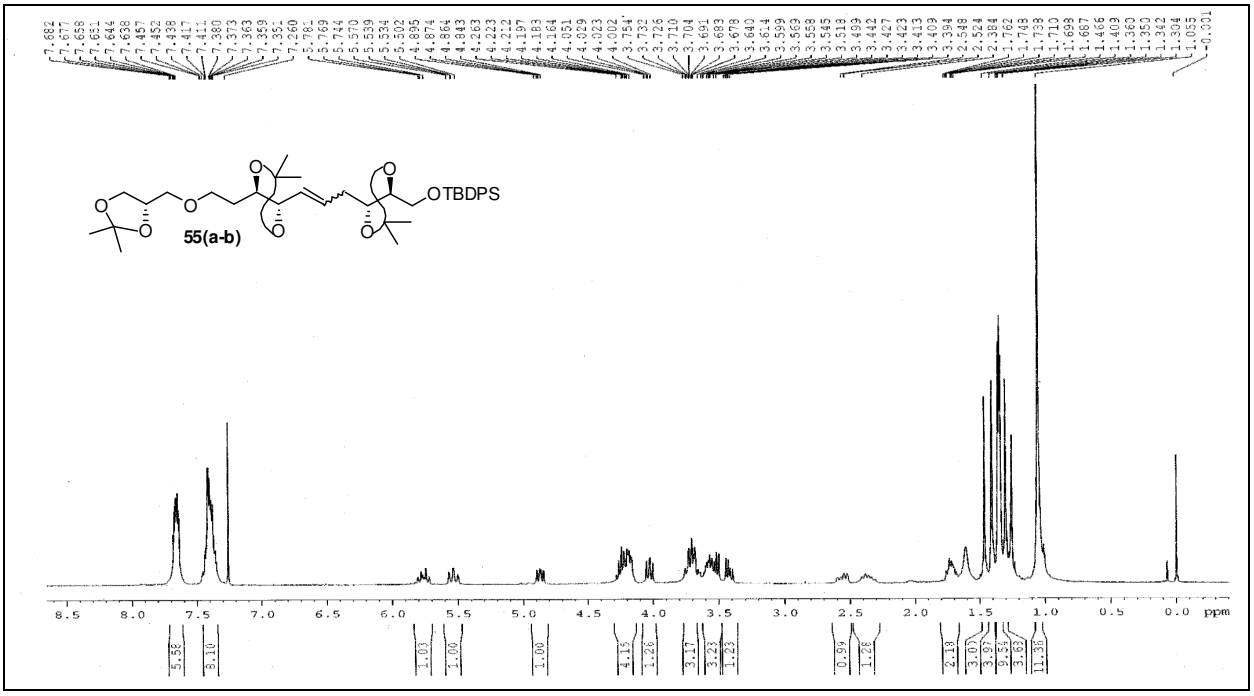
^{1}H NMR spectrum of 46 (300 MHz, $CDCl_3$):



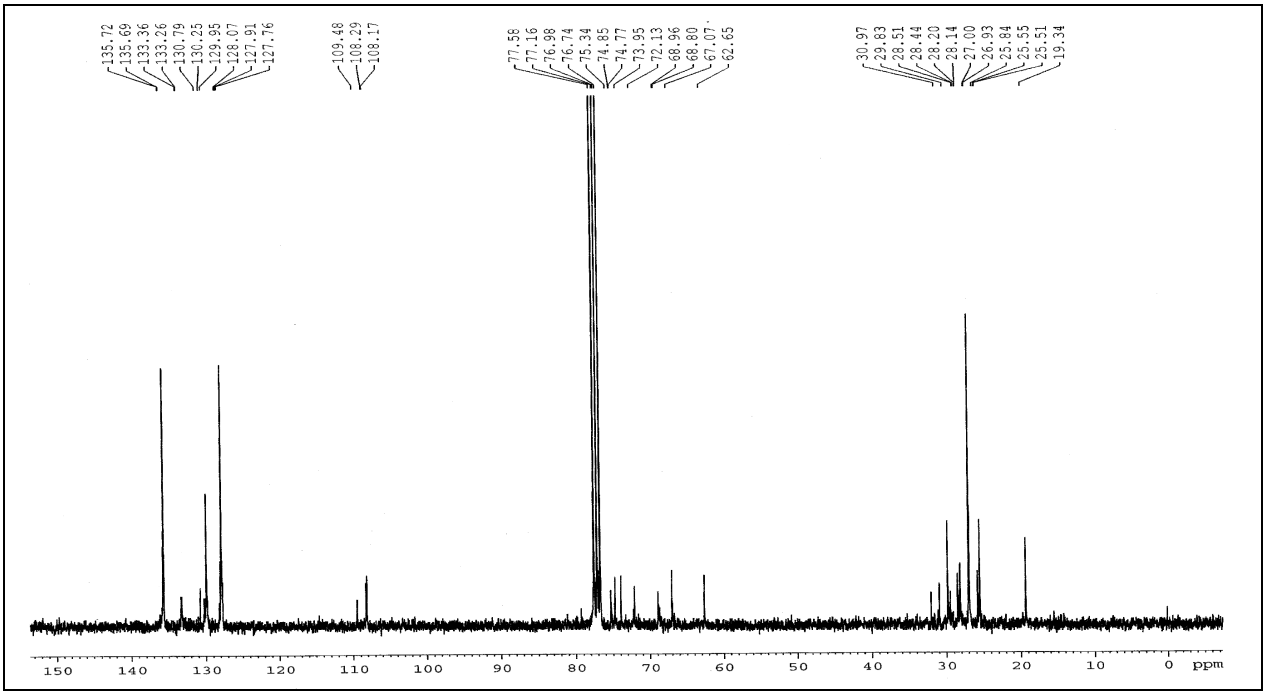
^{13}C NMR spectrum of 46 (75 MHz, $CDCl_3$):



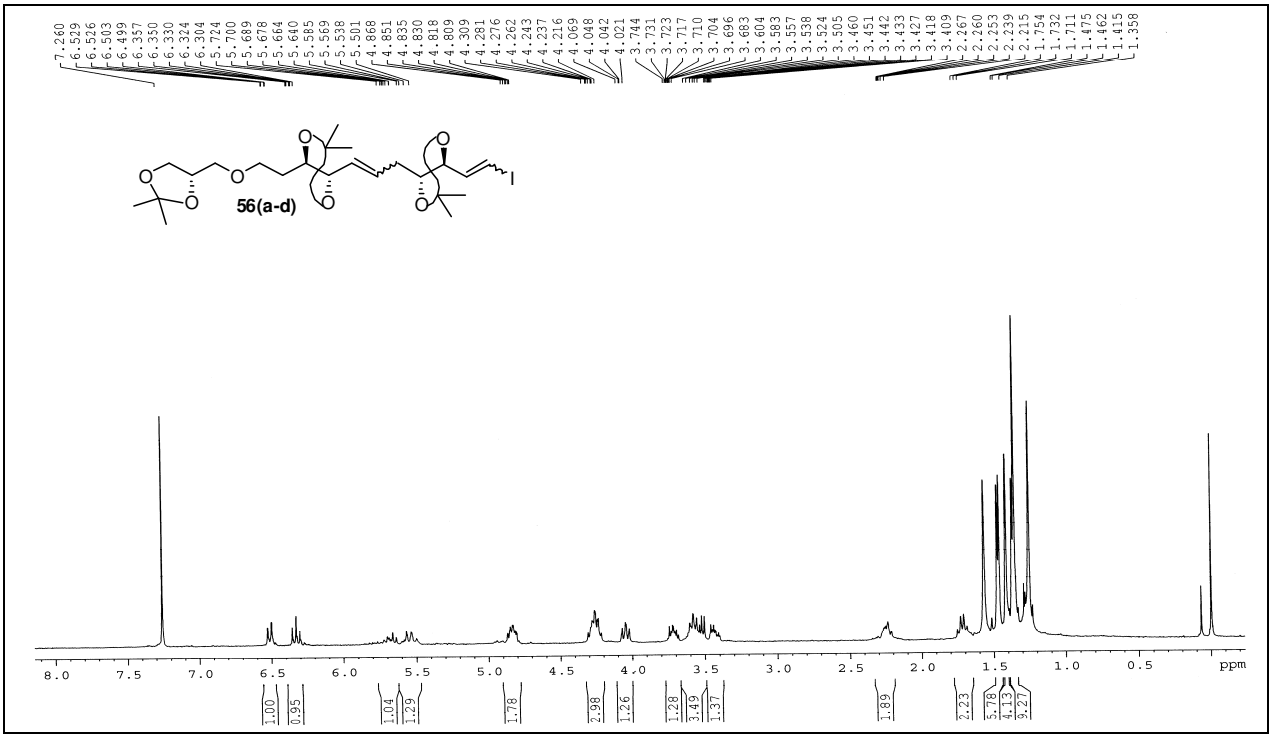
^{1}H NMR spectrum of 55 (a-b) (300 MHz, $CDCl_3$):



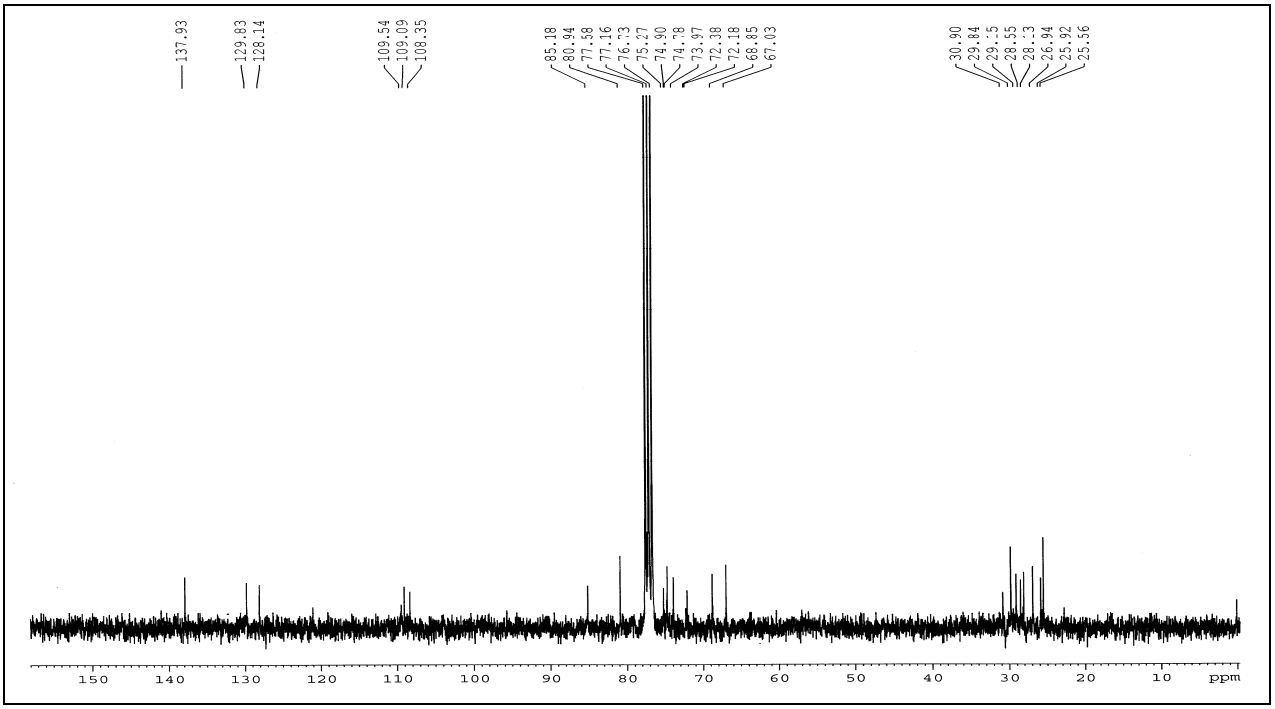
^{13}C NMR spectrum of 55 (a-b) (75 MHz, $CDCl_3$):



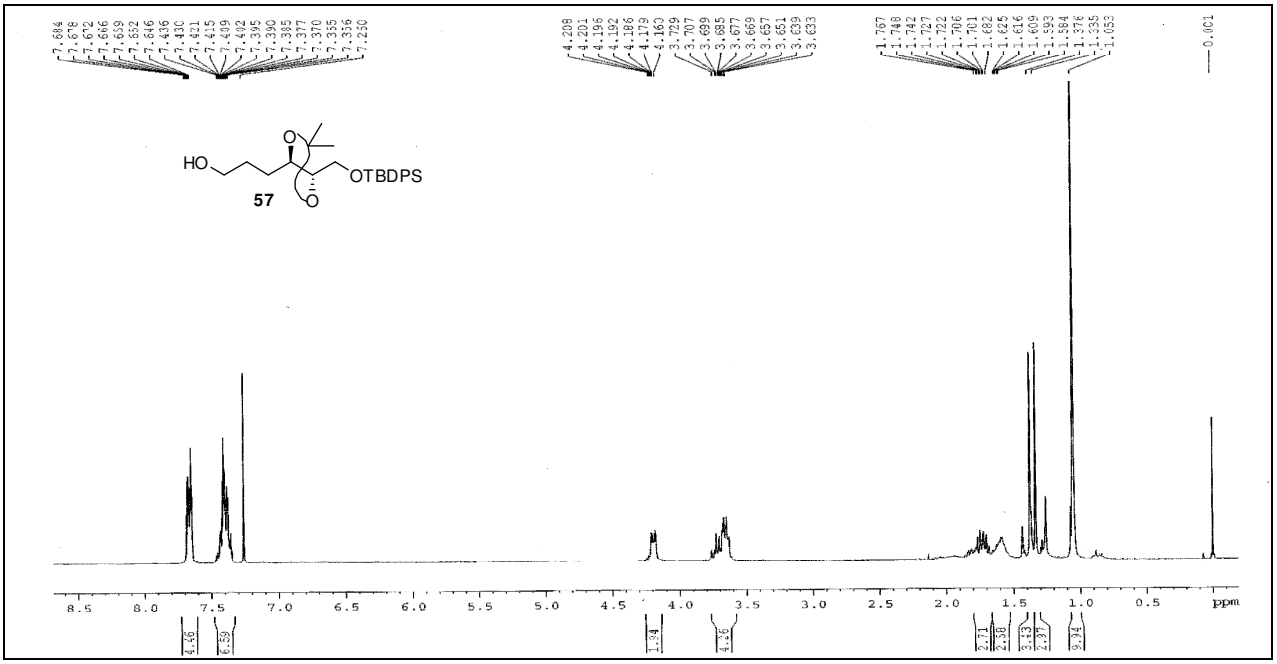
^{1}H NMR spectrum of 56 (a-d) (300 MHz, $CDCl_3$):



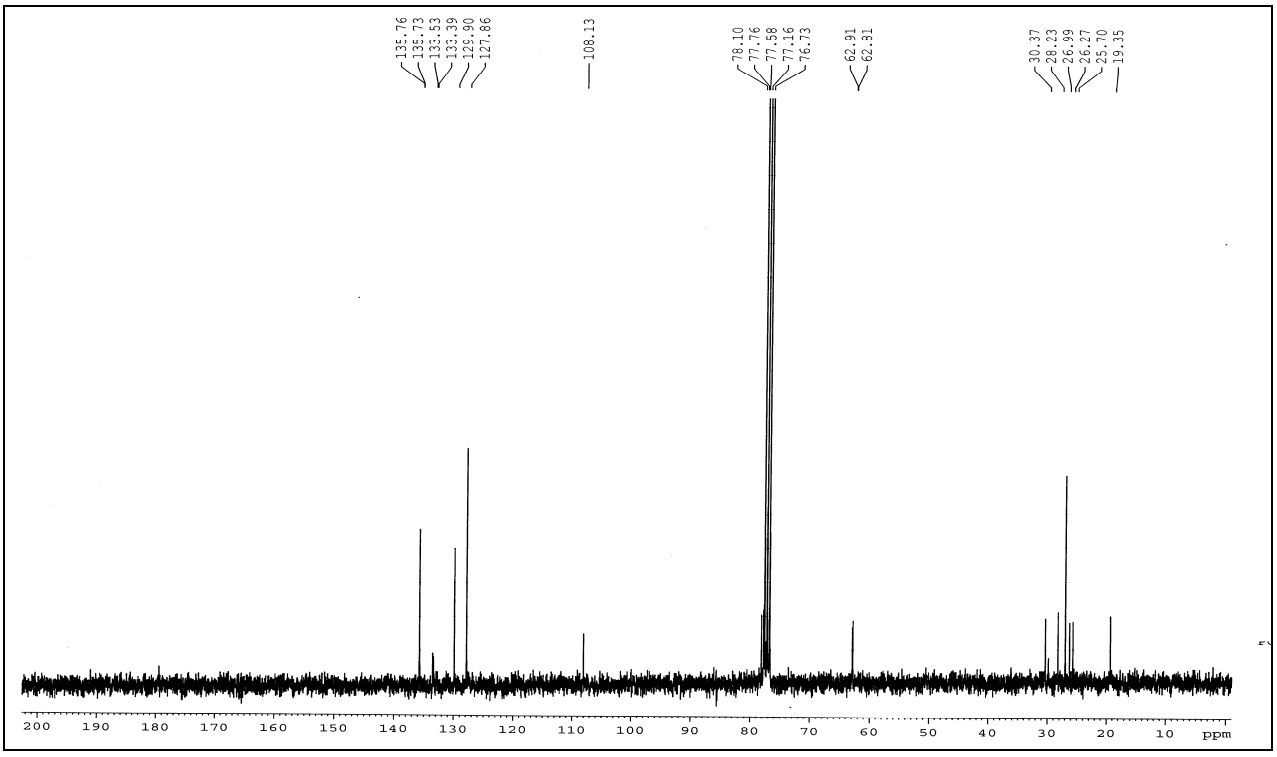
^{13}C NMR spectrum of 56 (a-d) (75 MHz, $CDCl_3$):



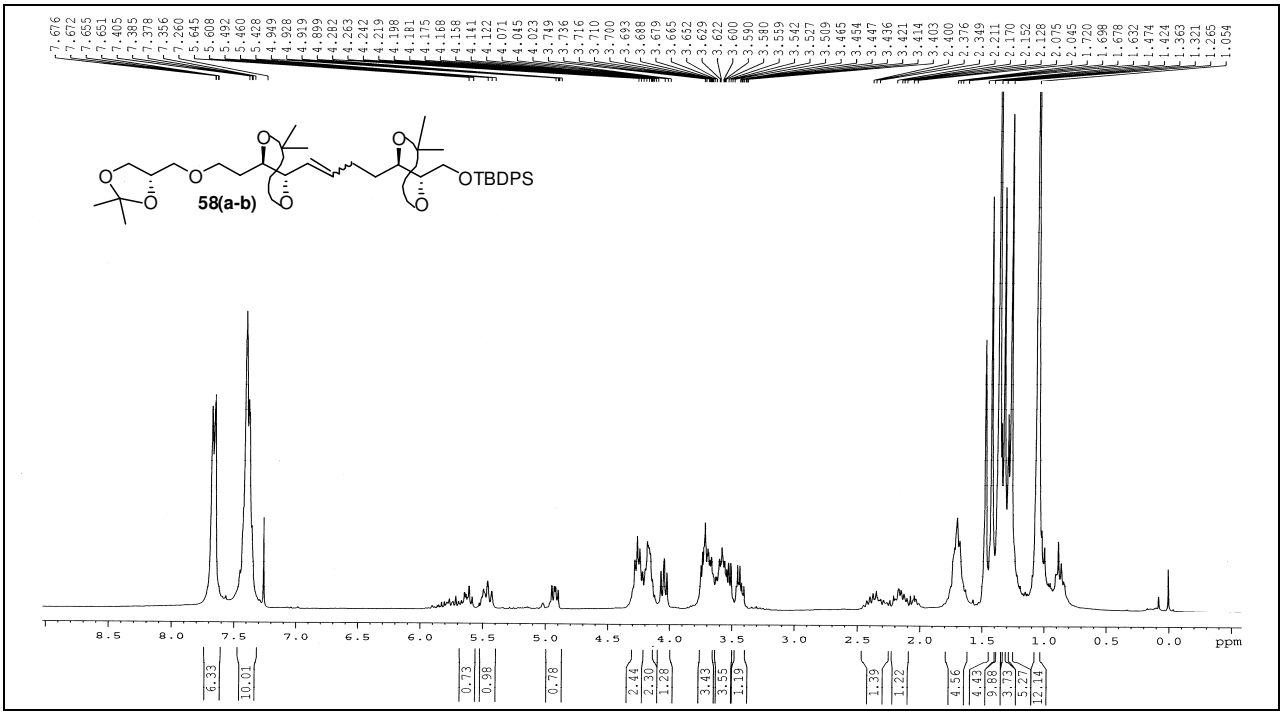
^{1}H NMR spectrum of 57 (300 MHz, $CDCl_3$):



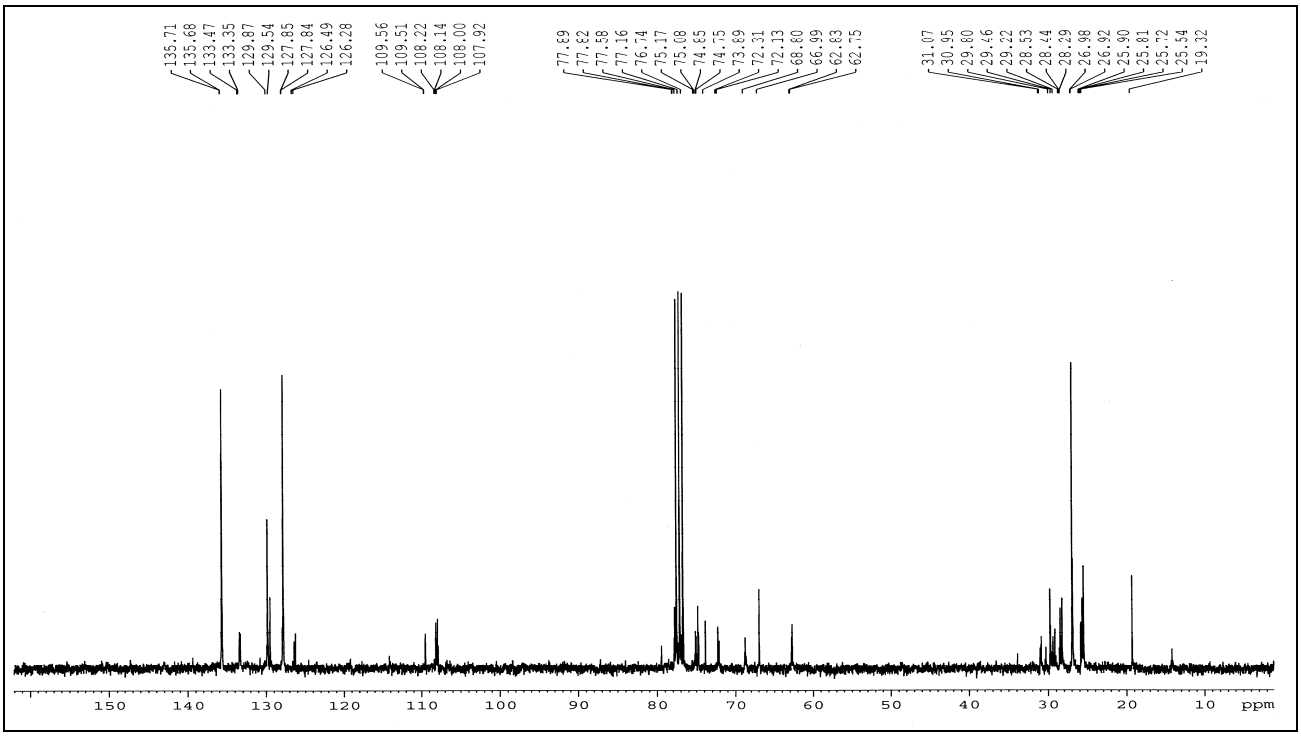
^{13}C NMR spectrum of 57 (75 MHz, $CDCl_3$):



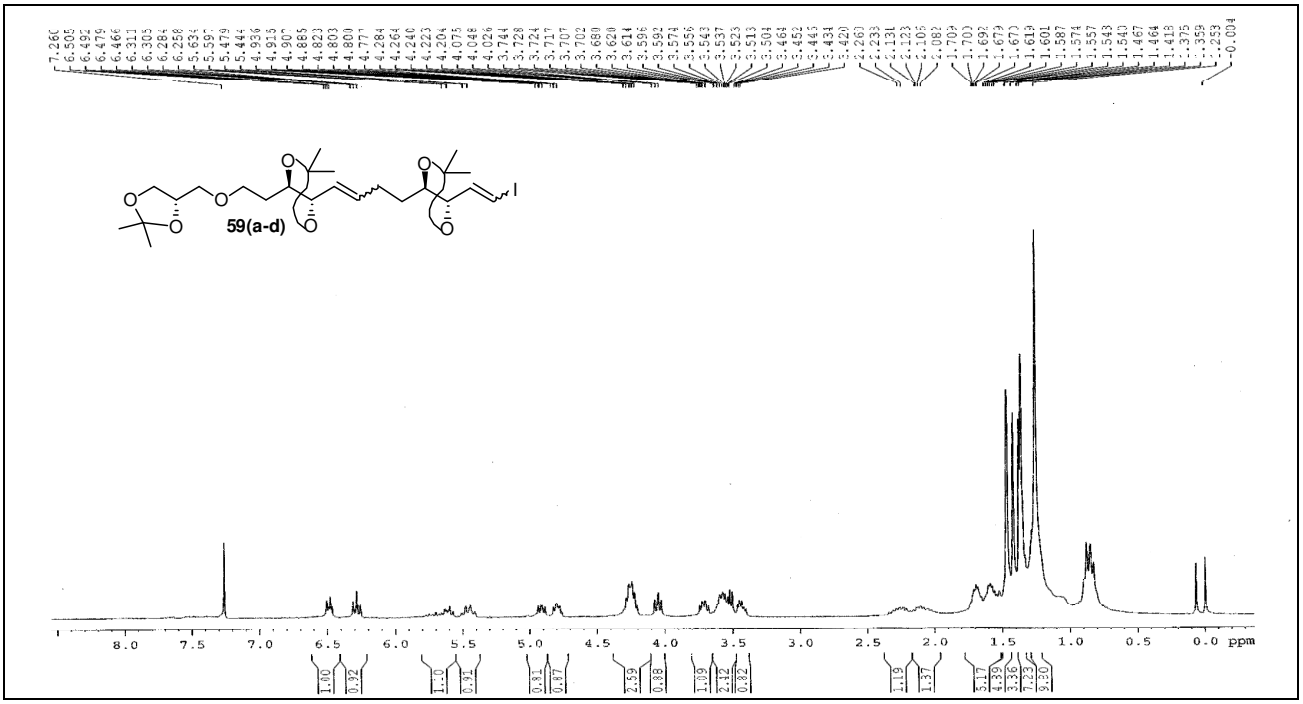
^{1}H NMR spectrum of 58 (a-b) (300 MHz, $CDCl_3$):



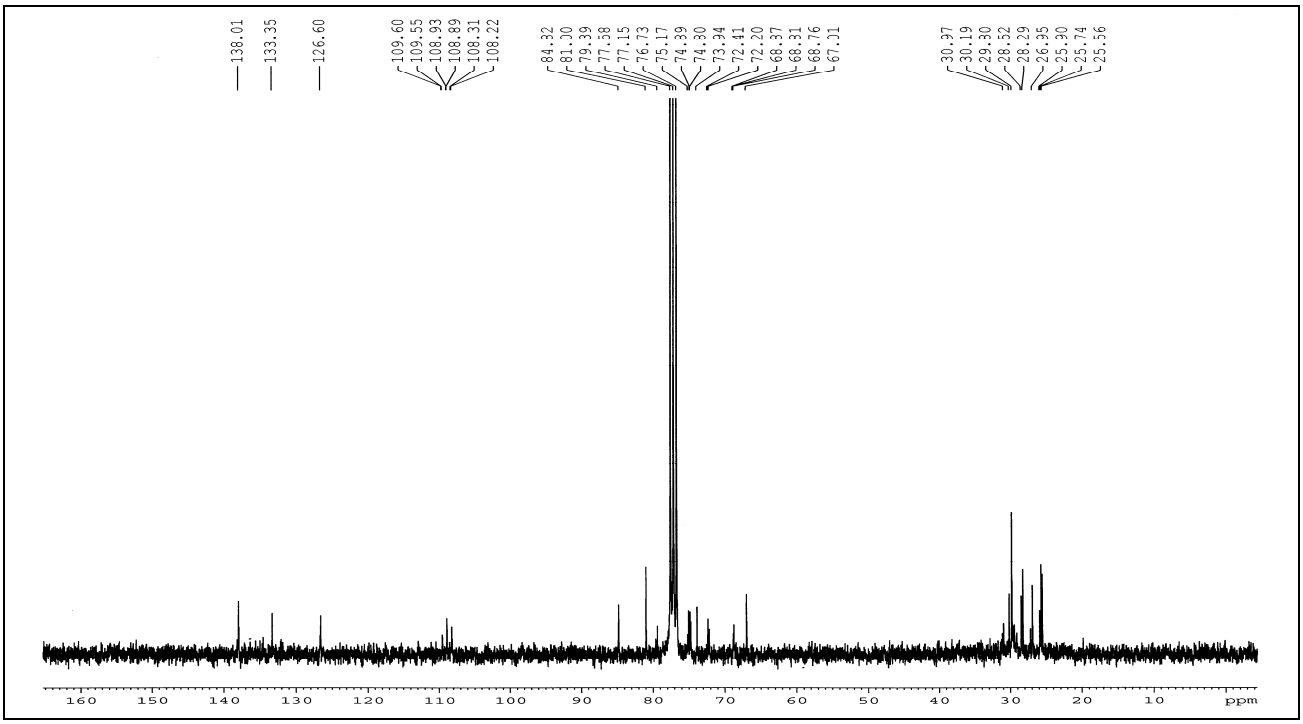
^{13}C NMR spectrum of 58 (a-b) (75 MHz, $CDCl_3$):



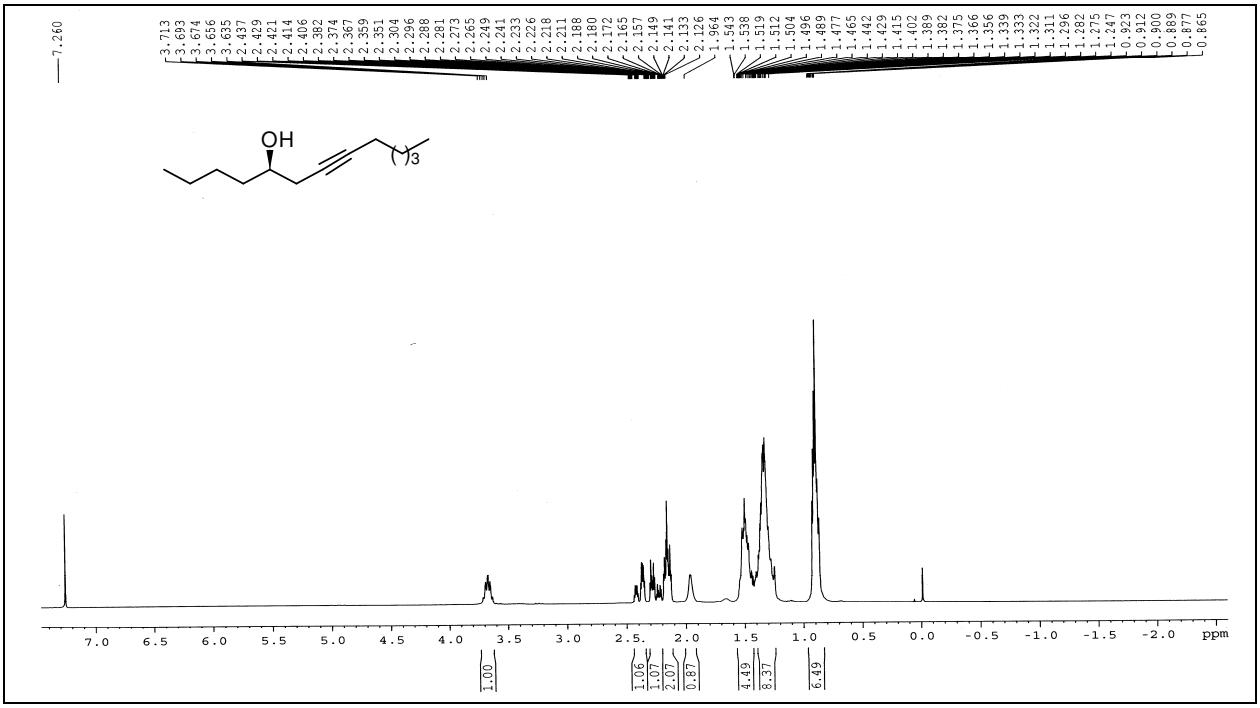
^{1}H NMR spectrum of 59 (a-d) (300 MHz, $CDCl_3$):



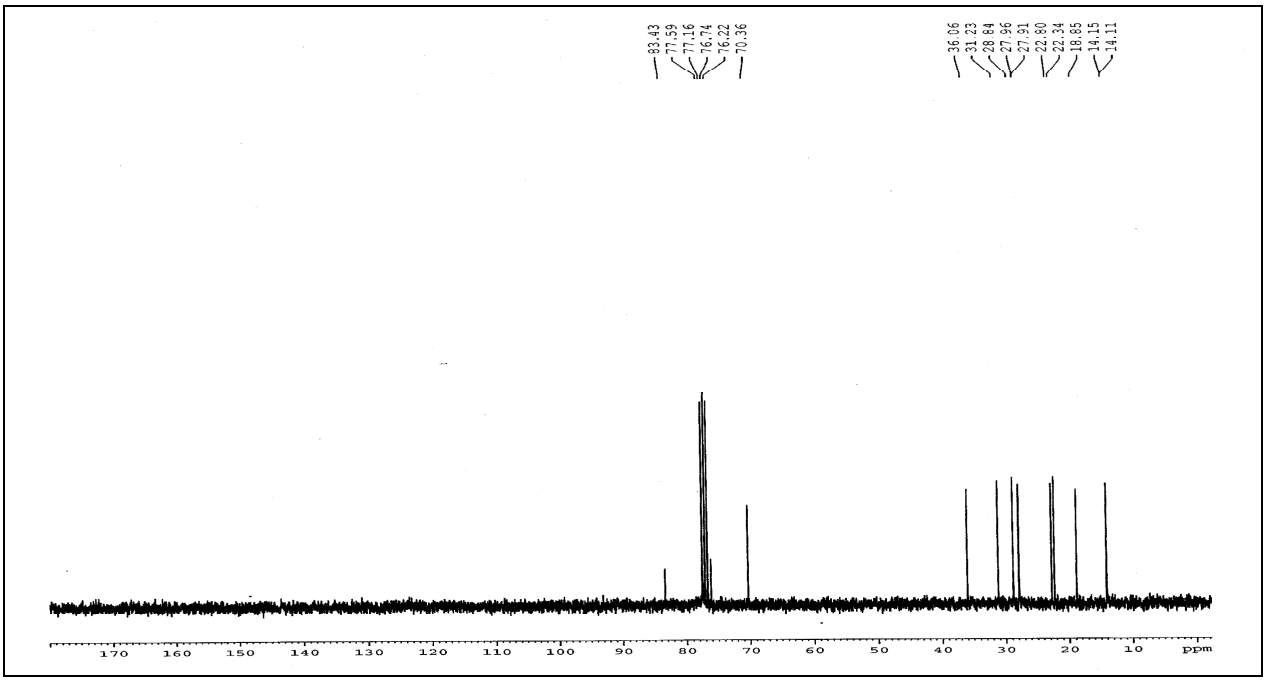
^{13}C NMR spectrum of 59 (a-b) (75 MHz, $CDCl_3$):



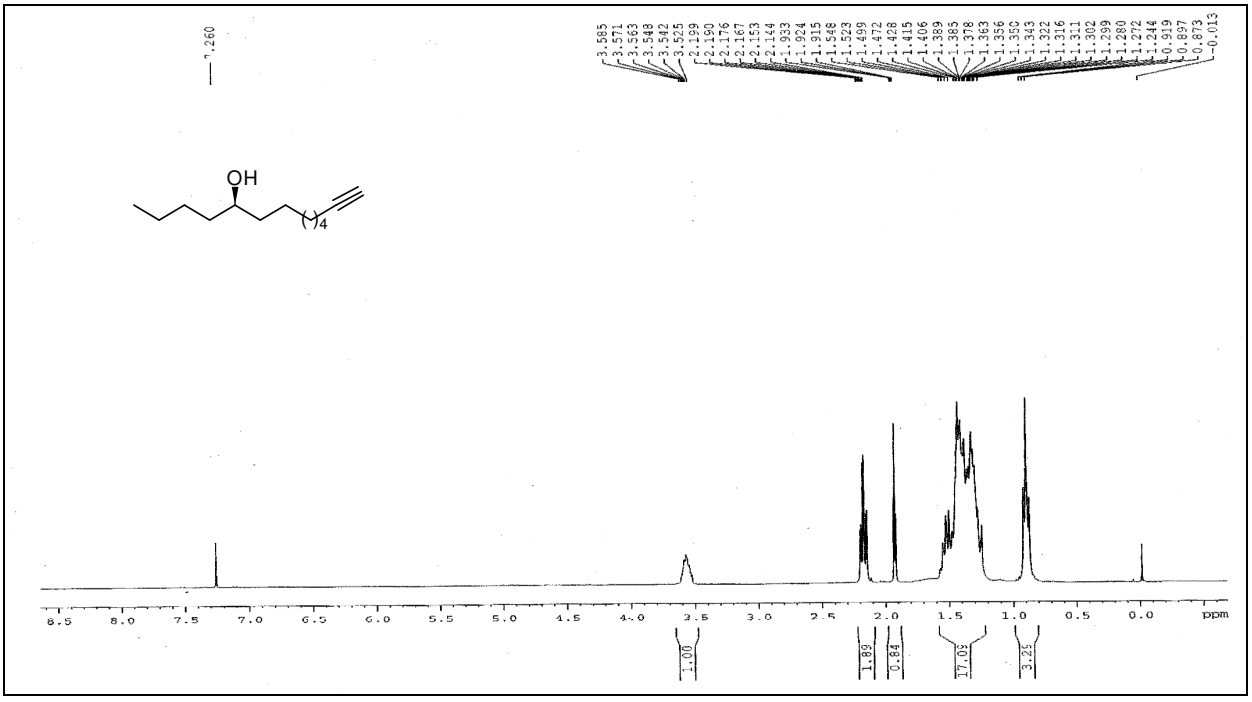
^{1}H NMR spectrum of 60a (300 MHz, $CDCl_3$):



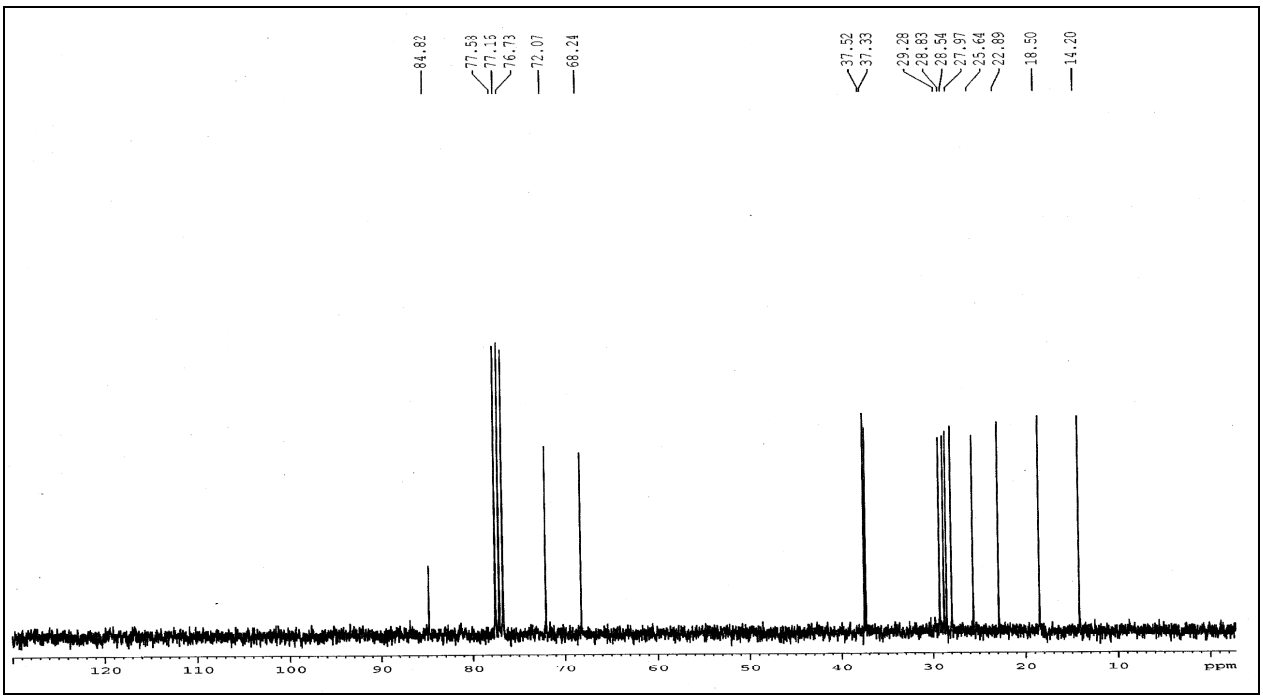
^{13}C NMR spectrum of 60a (75 MHz, $CDCl_3$):



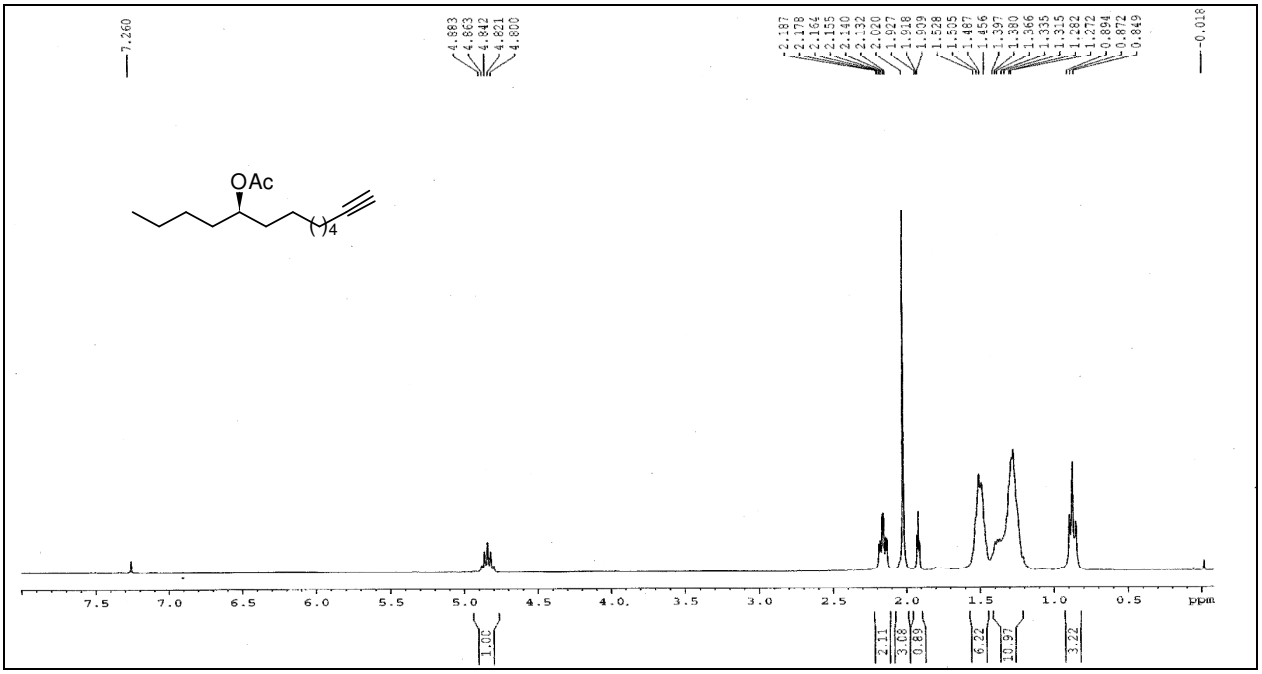
^{1}H NMR spectrum of 61a (300 MHz, $CDCl_3$):



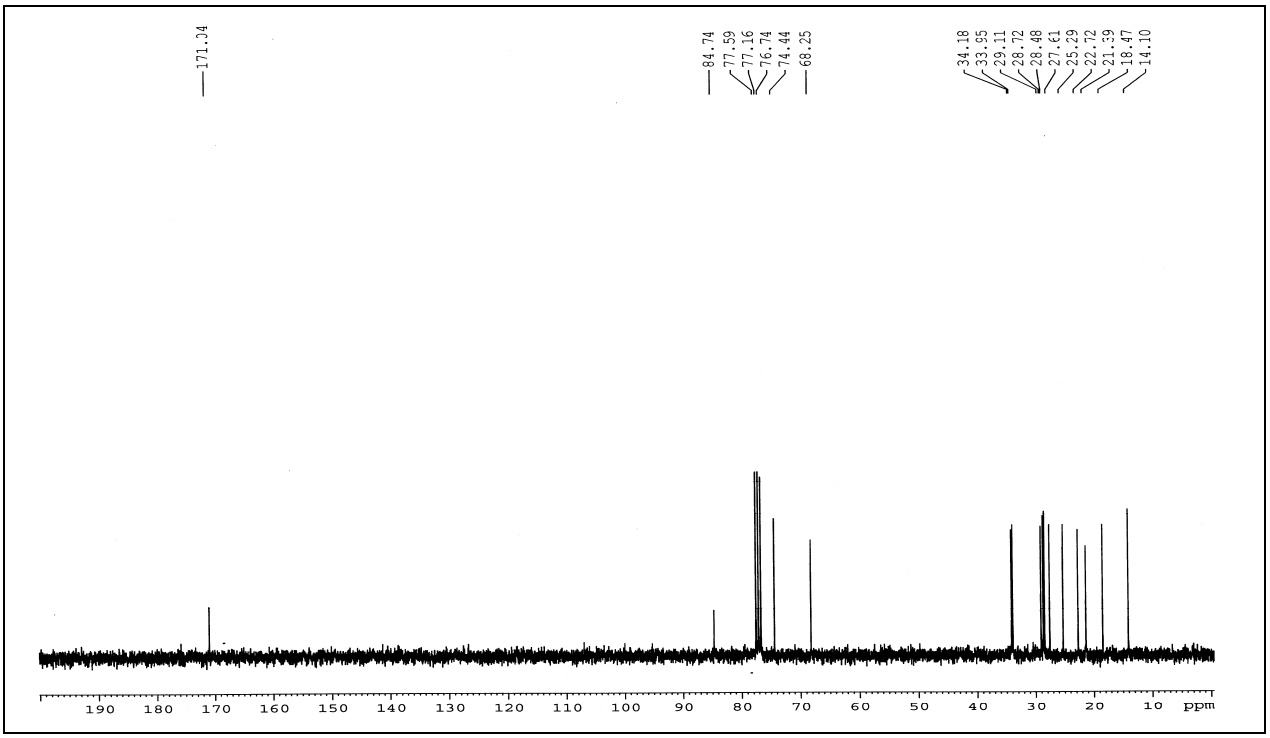
^{13}C NMR spectrum of 61a (75 MHz, $CDCl_3$):



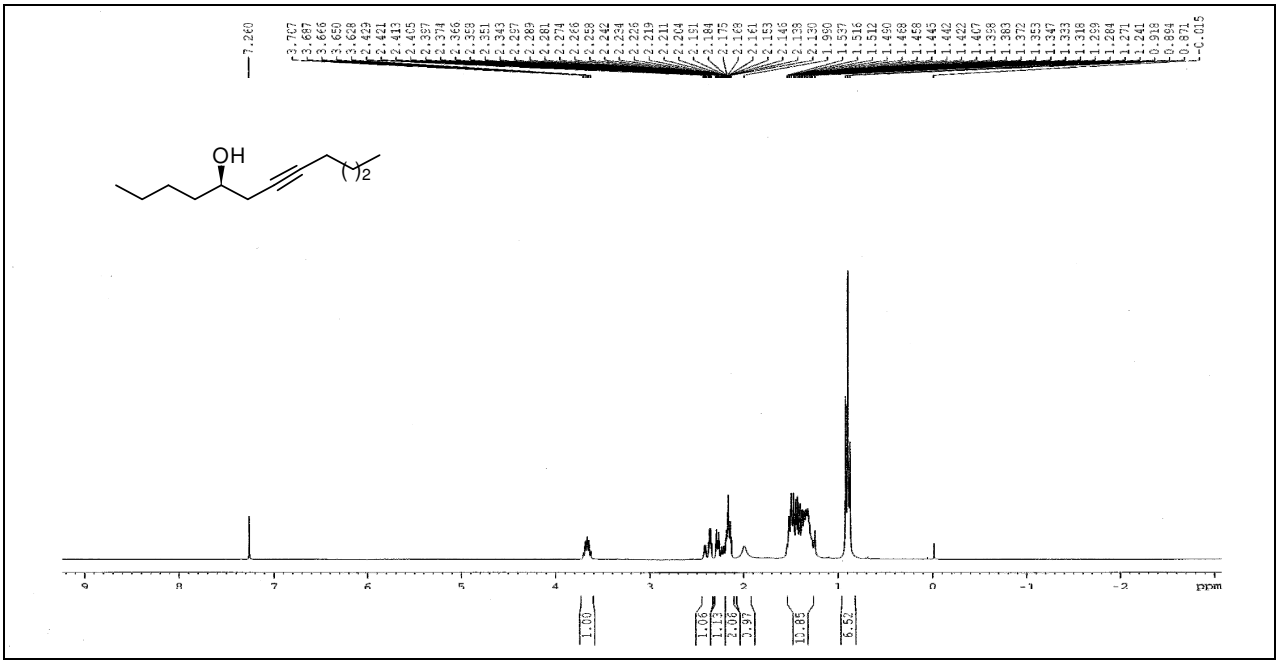
^{1}H NMR spectrum of 62a (300 MHz, $CDCl_3$):



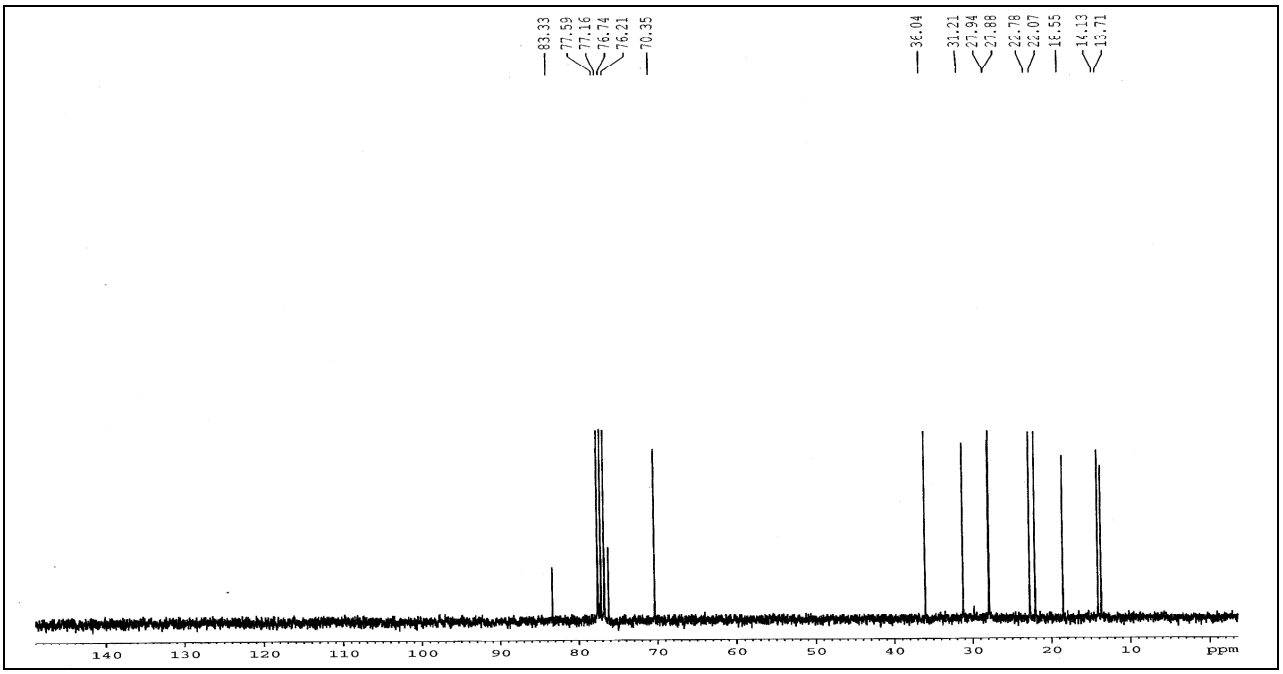
^{13}C NMR spectrum of 62a (75 MHz, $CDCl_3$):



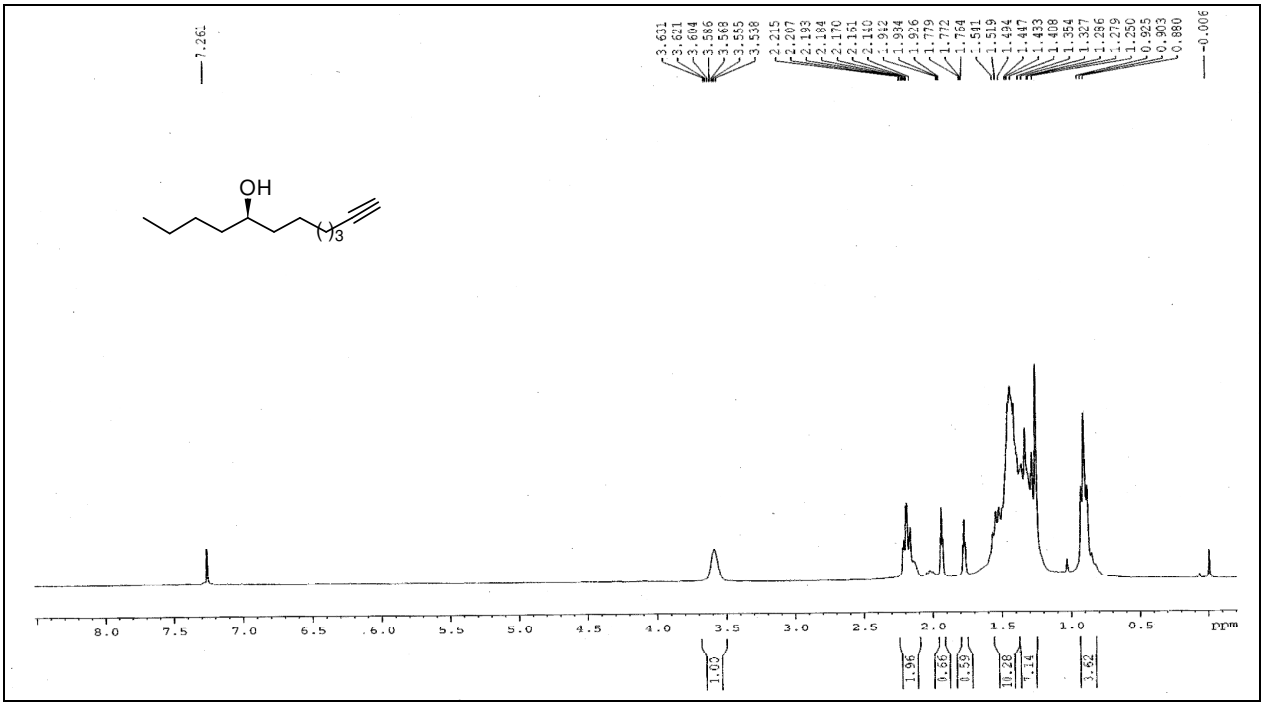
^{1}H NMR spectrum of 60b (300 MHz, $CDCl_3$):



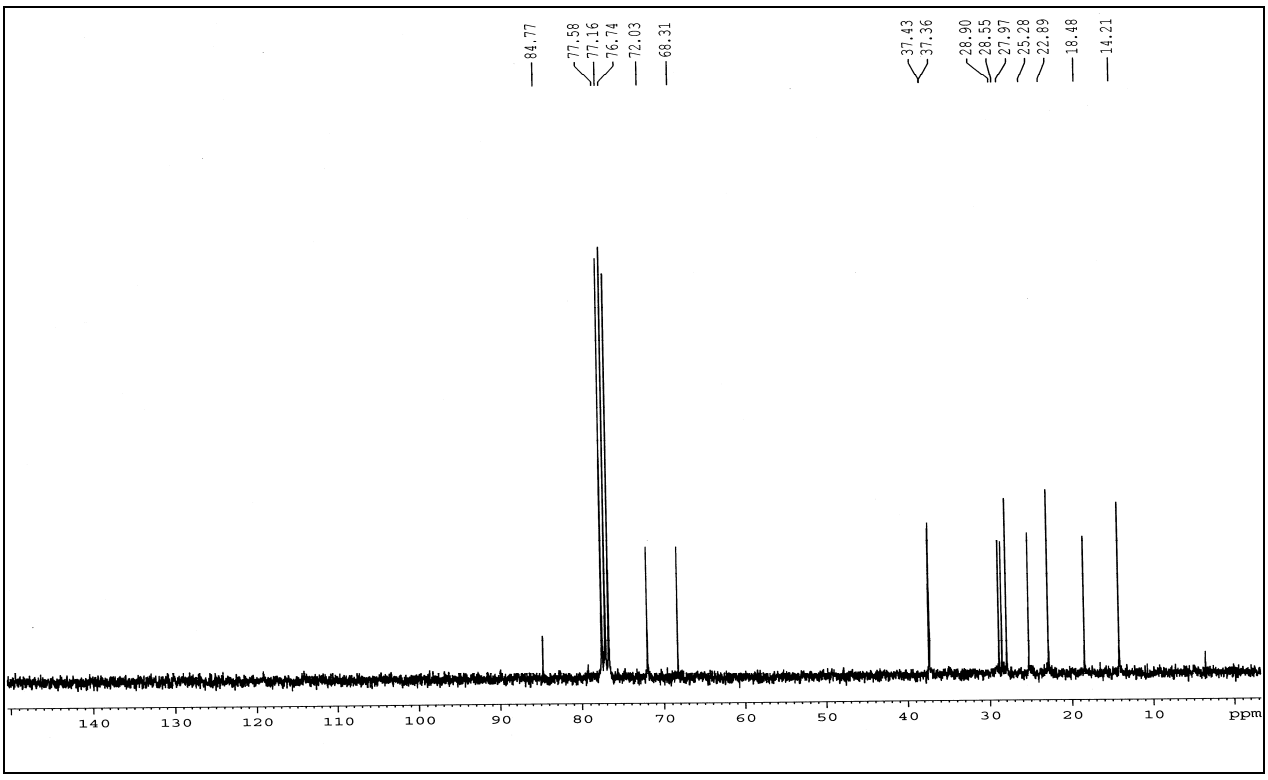
^{13}C NMR spectrum of 60b (75 MHz, $CDCl_3$):



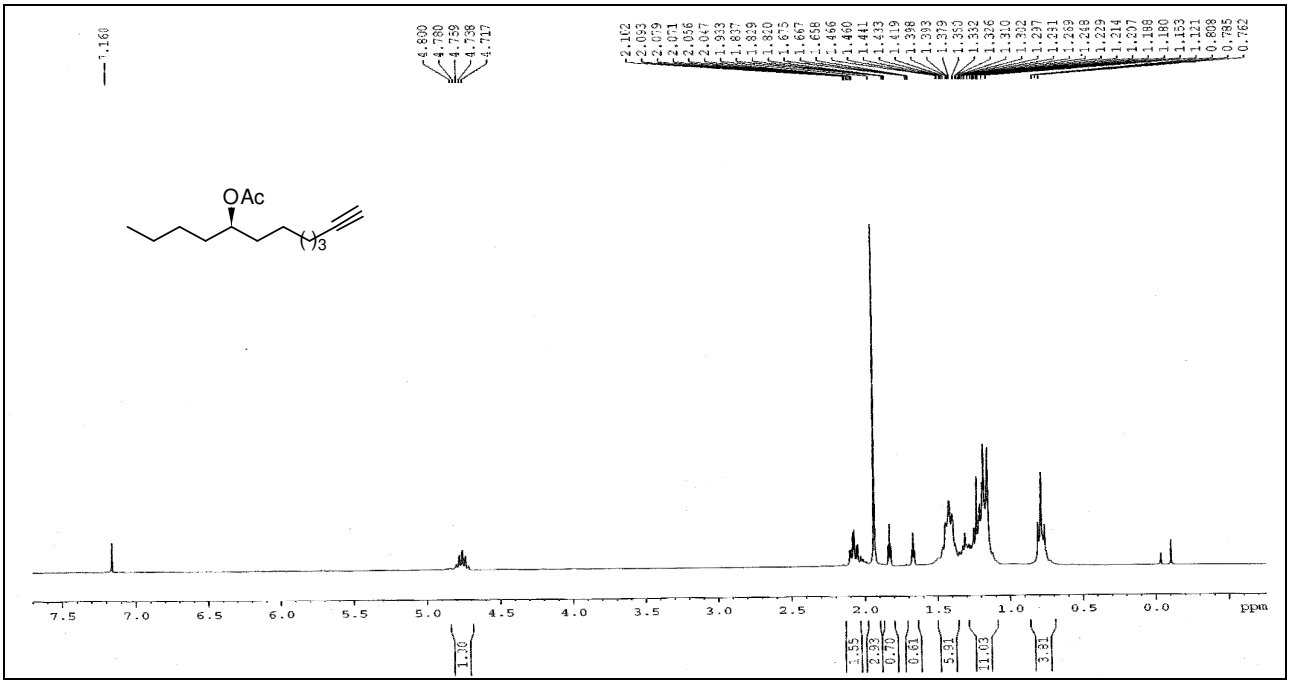
^{1}H NMR spectrum of 61b (300 MHz, $CDCl_3$):



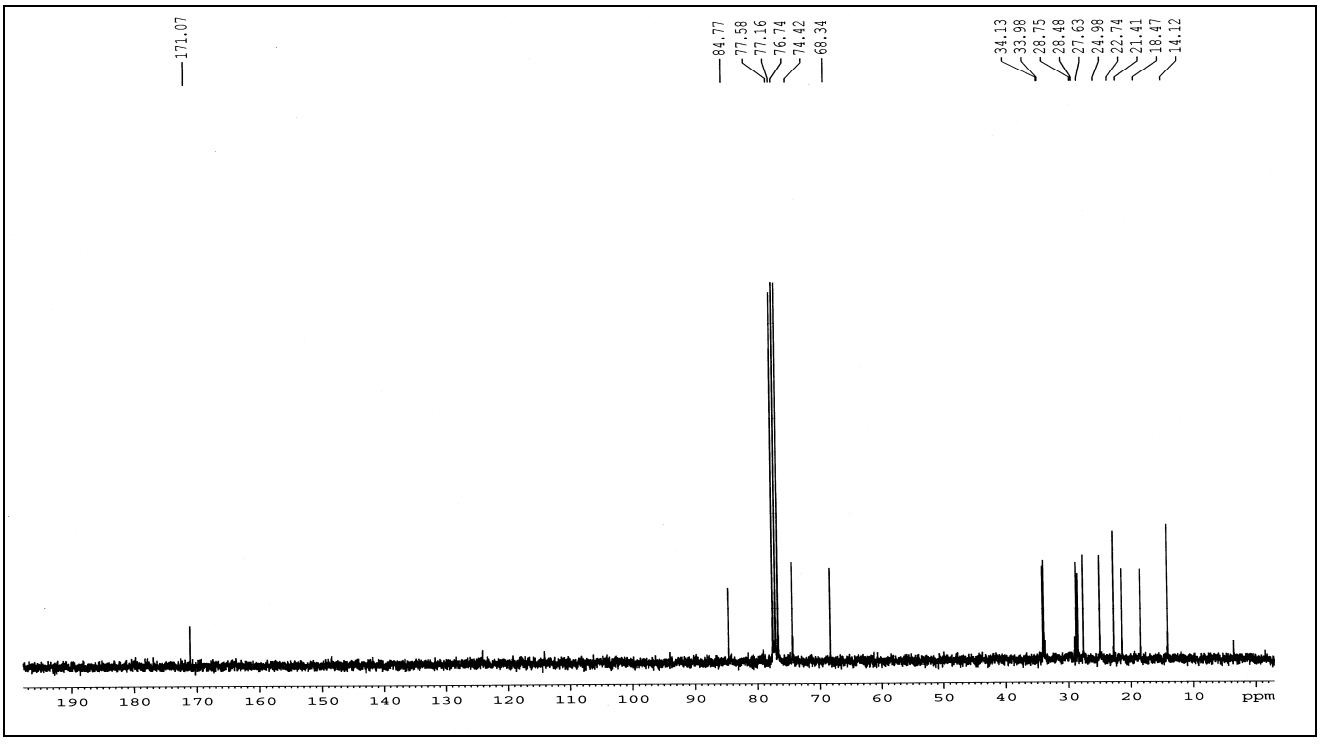
^{13}C NMR spectrum of 61b (75 MHz, $CDCl_3$):



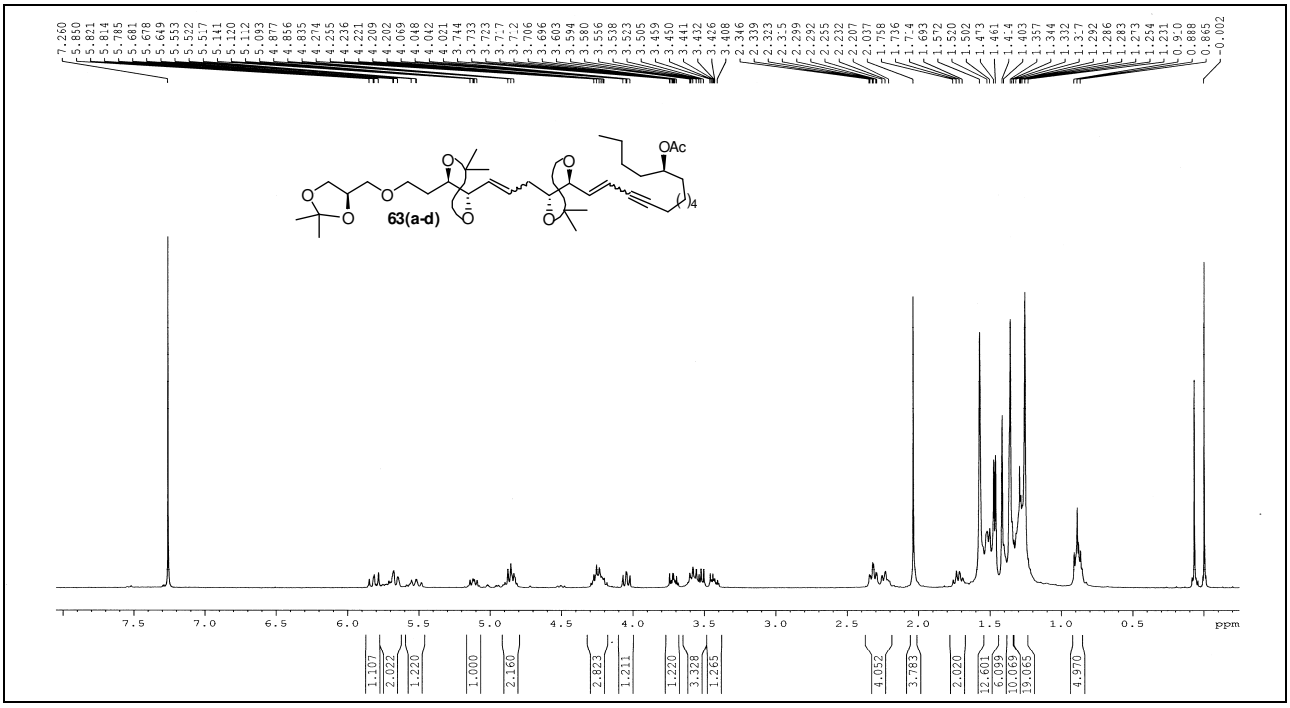
^{1}H NMR spectrum of 62b (300 MHz, $CDCl_3$):



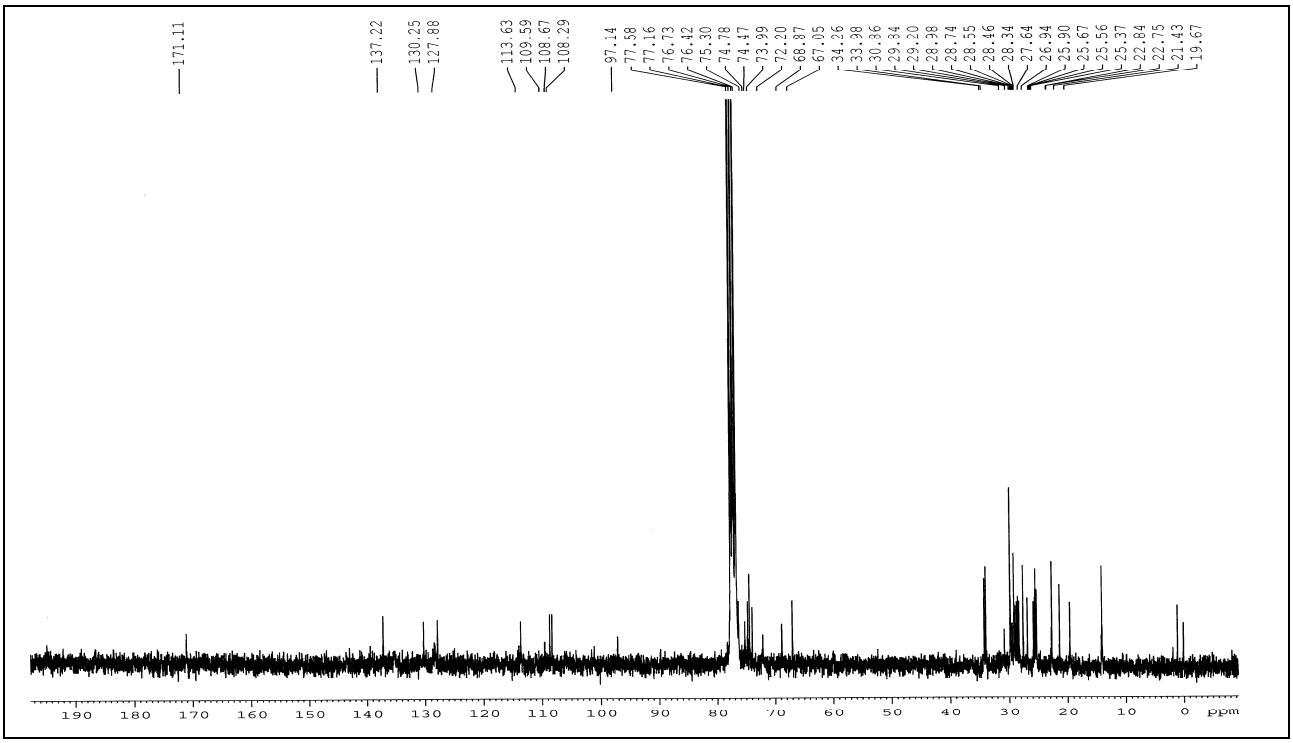
^{13}C NMR spectrum of 62b (75 MHz, $CDCl_3$):



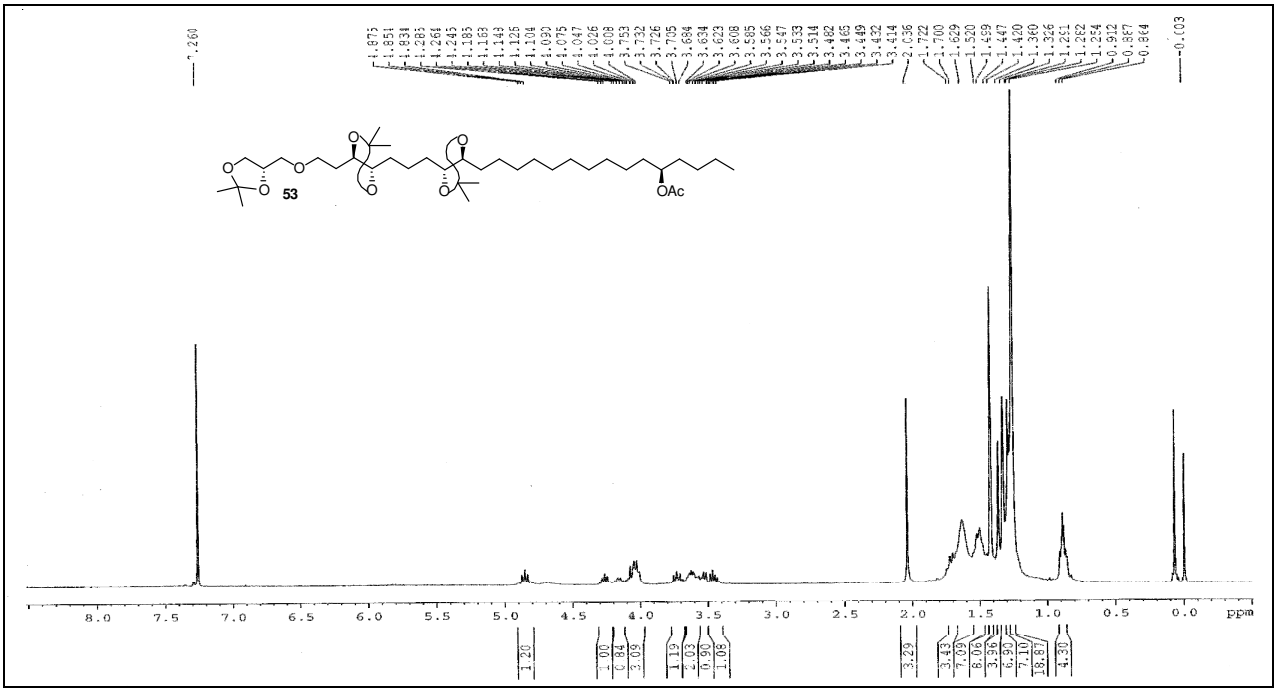
^{1}H NMR spectrum of 63(a-d) (300 MHz, $CDCl_3$):



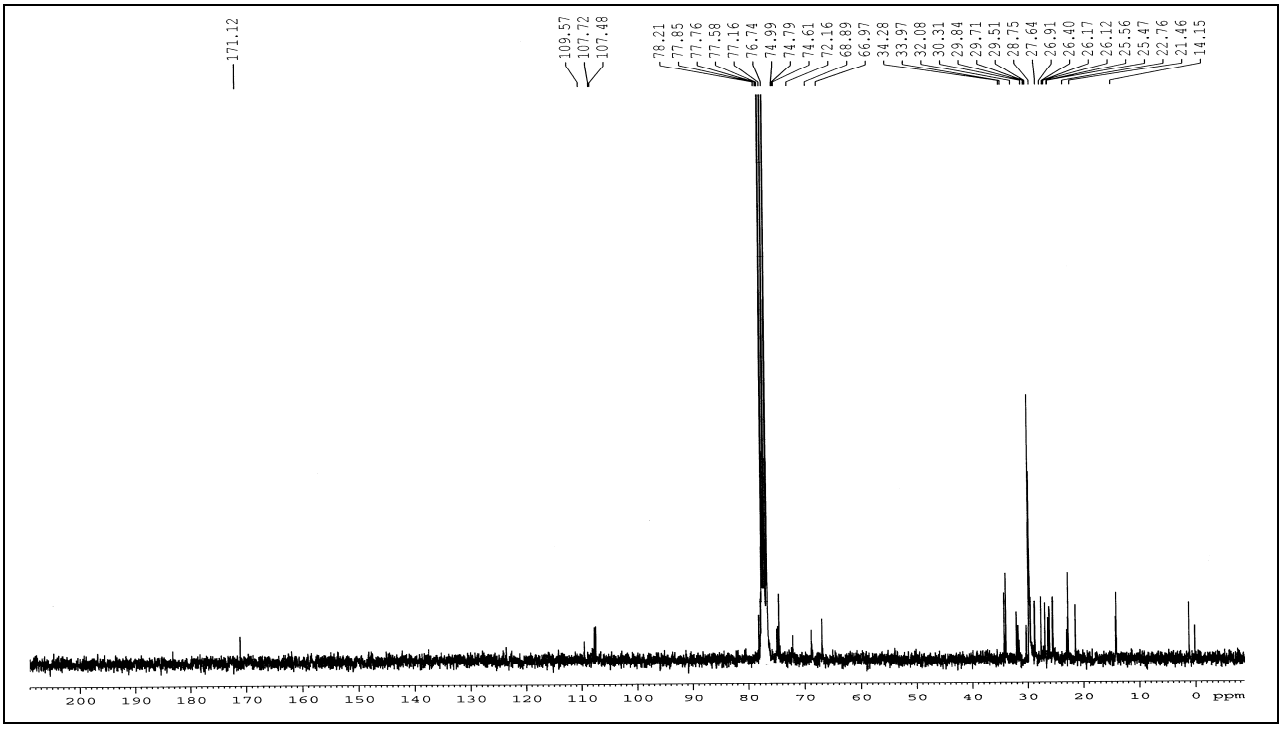
^{13}C NMR spectrum of 63(a-d) (75 MHz, $CDCl_3$):



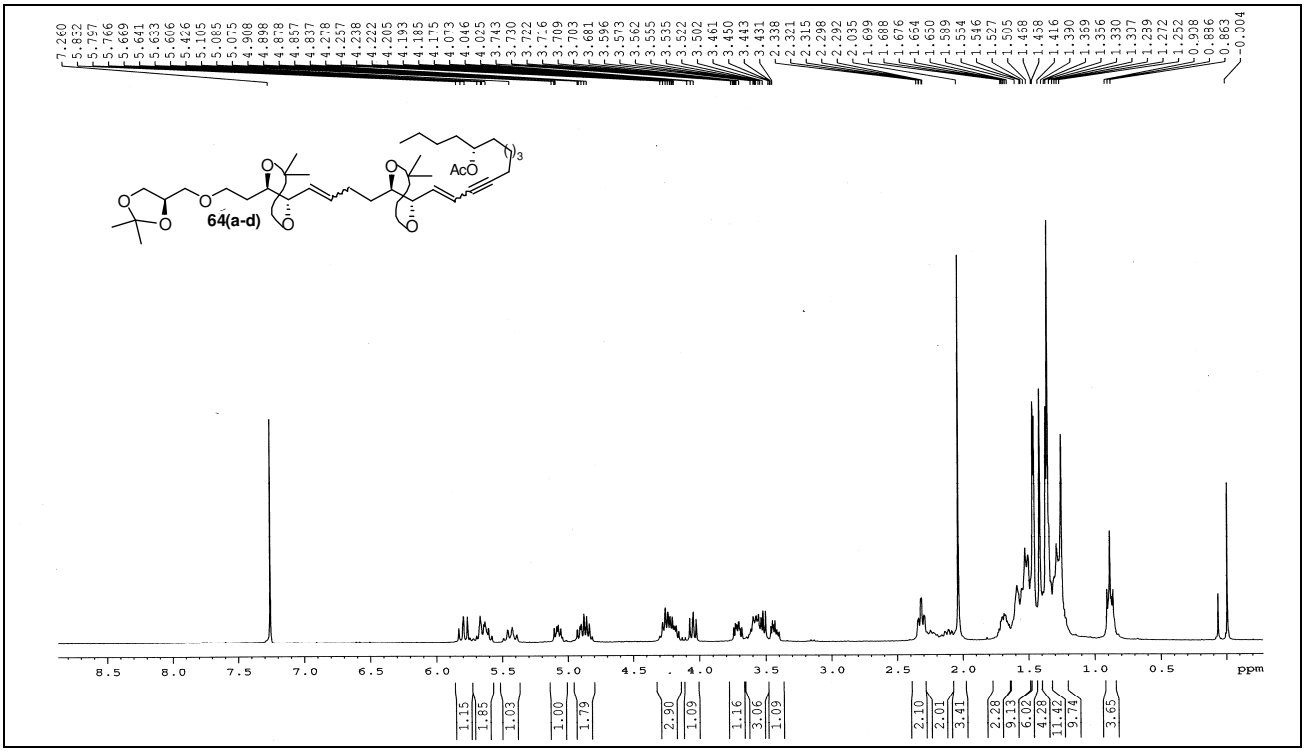
^{1}H NMR spectrum of 53 (300 MHz, $CDCl_3$):



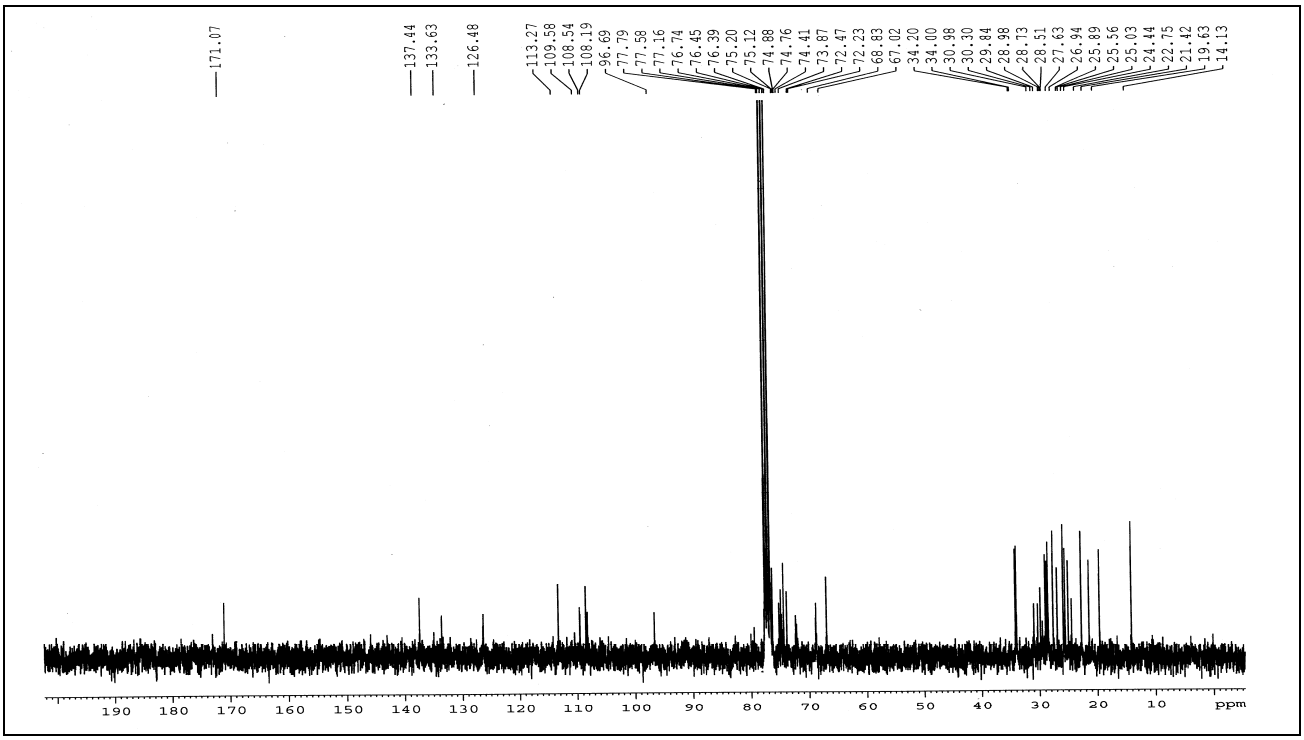
^{13}C NMR spectrum of 53 (75 MHz, $CDCl_3$):



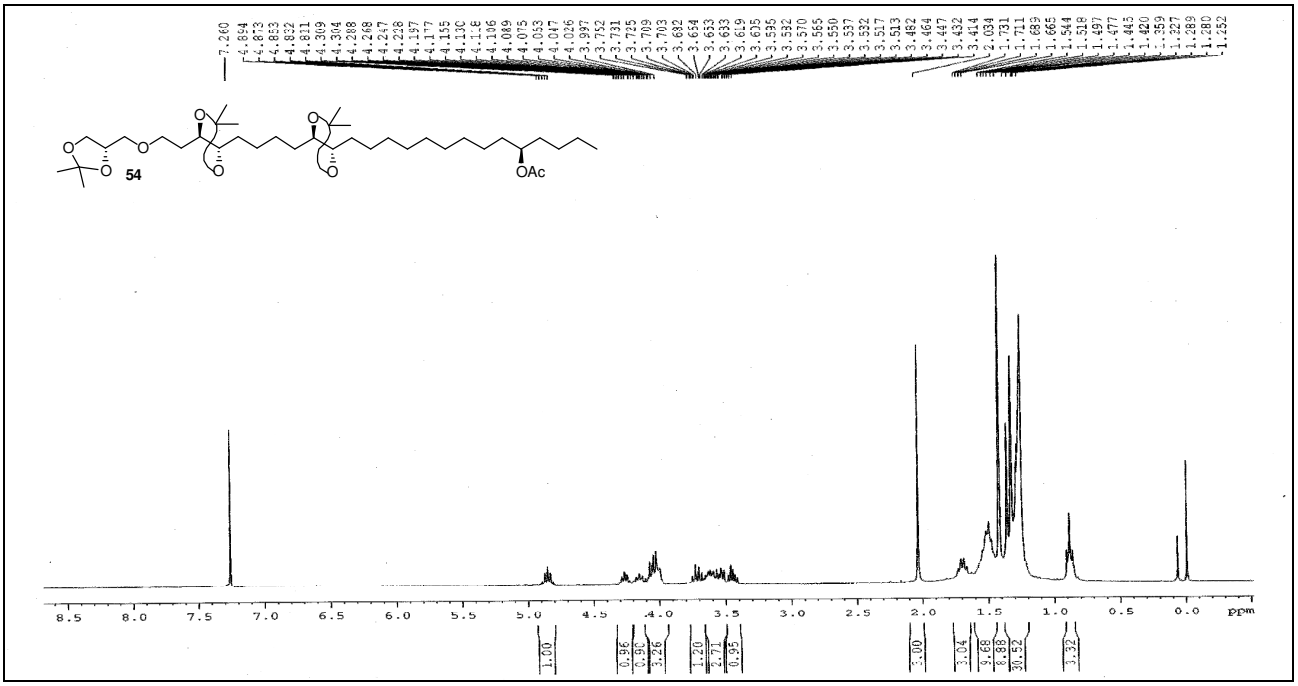
^{1}H NMR spectrum of 64 (a-d) (300 MHz, $CDCl_3$):



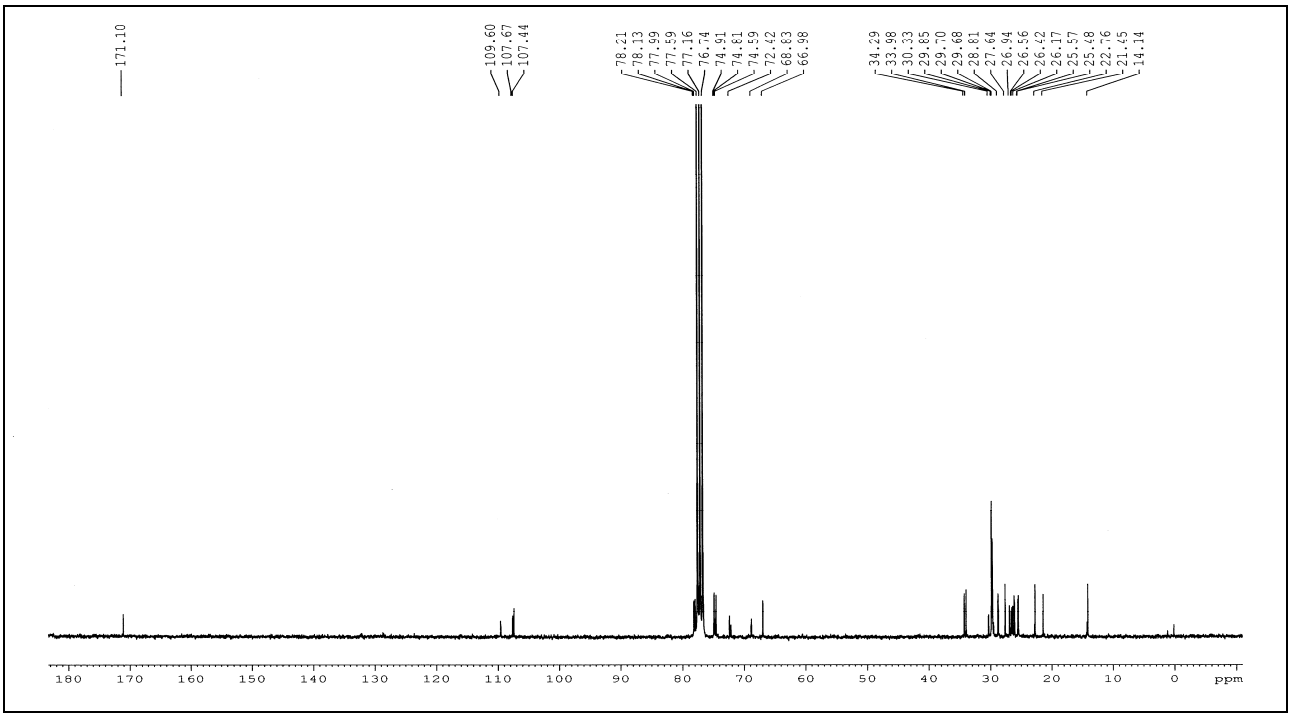
^{13}C NMR spectrum of 64 (a-d) (75 MHz, $CDCl_3$):



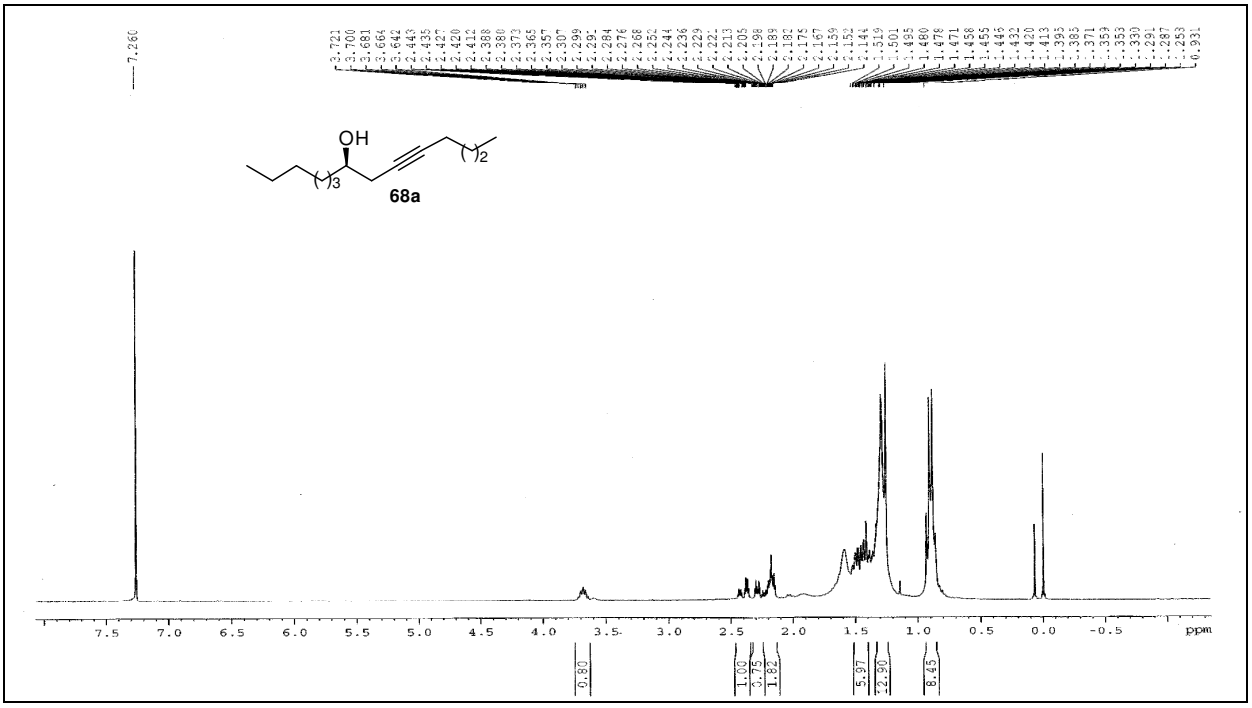
^{1}H NMR spectrum of 54 (300 MHz, $CDCl_3$):



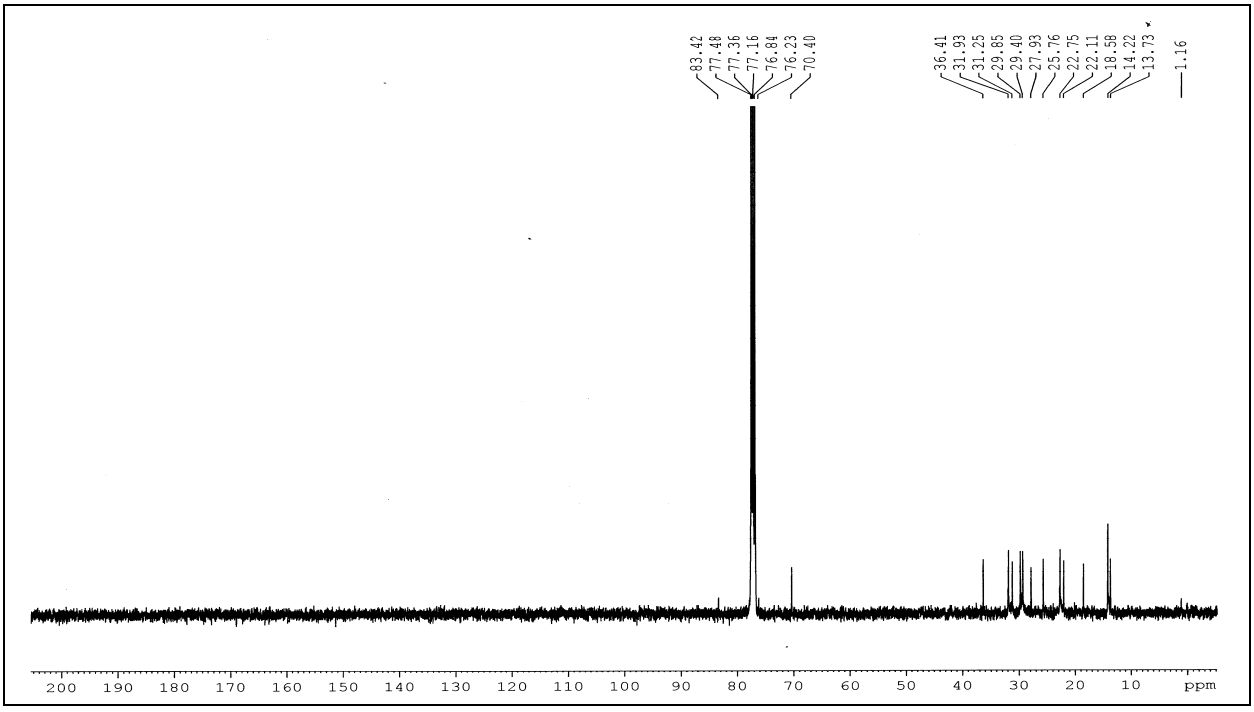
^{13}C NMR spectrum of 54 (75 MHz, $CDCl_3$):



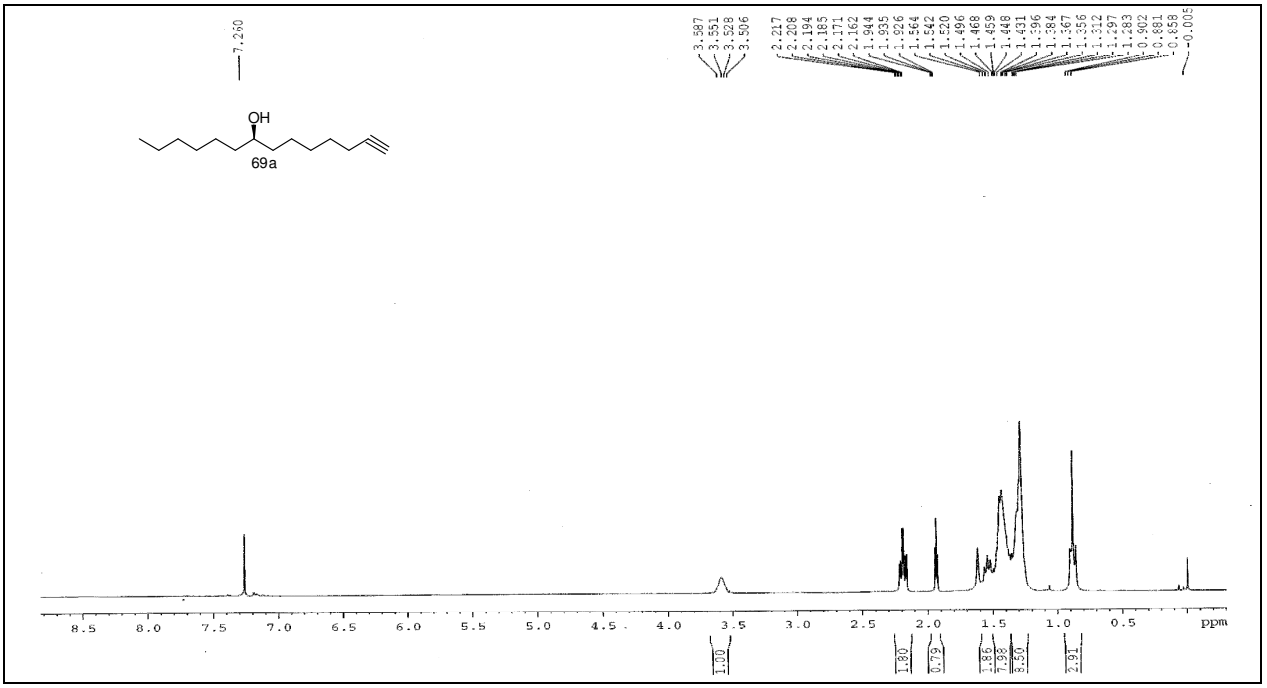
^{1}H NMR spectrum of 68a (300 MHz, $CDCl_3$):



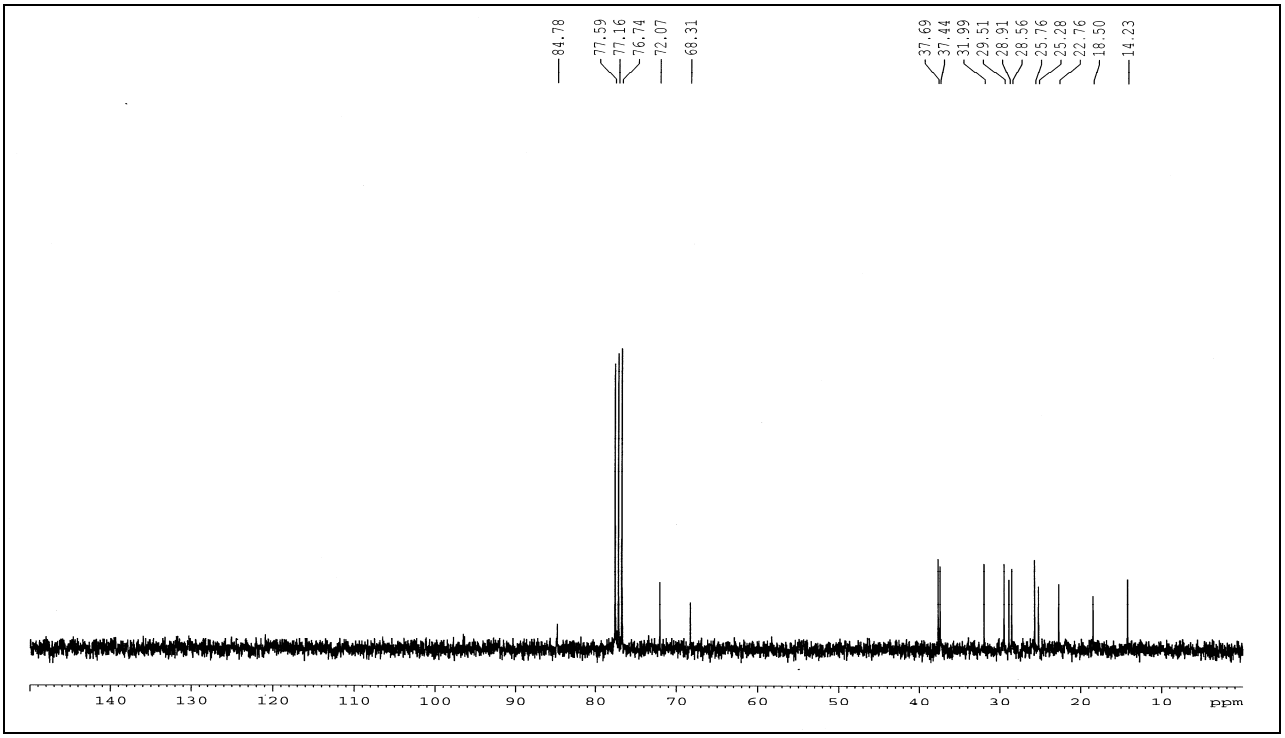
^{13}C NMR spectrum of 68a (75 MHz, $CDCl_3$):



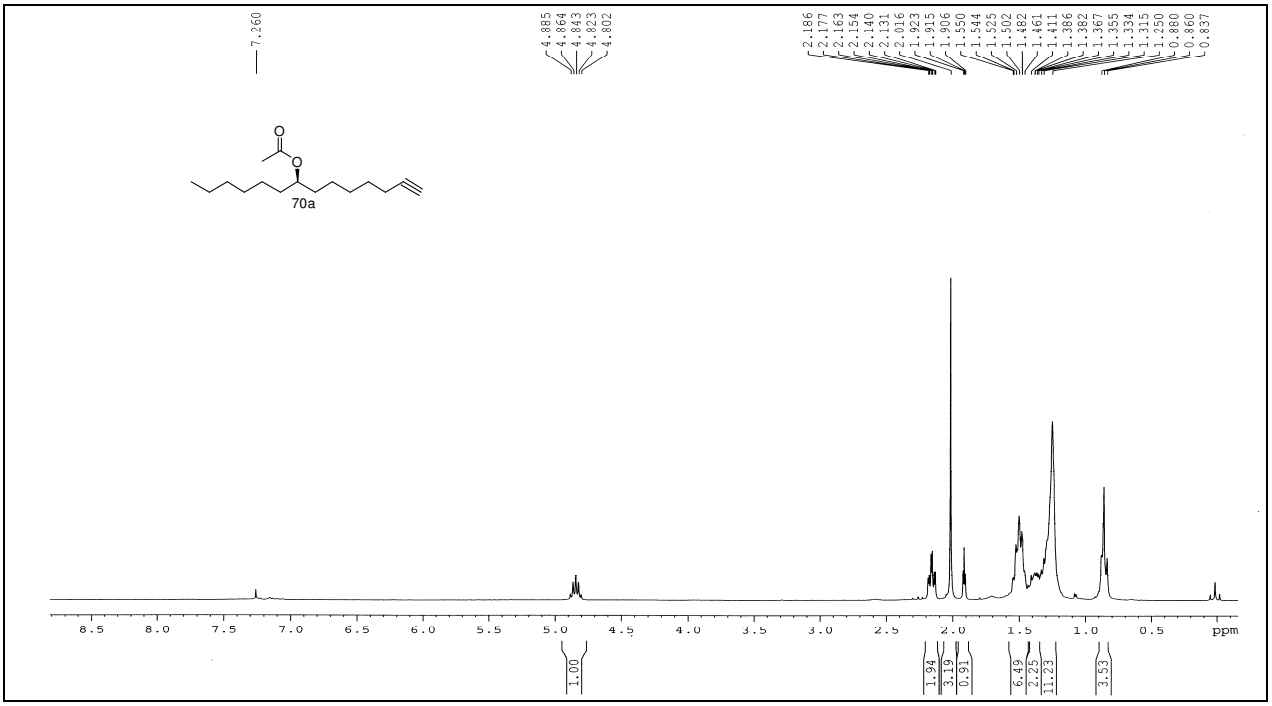
^{1}H NMR spectrum of 69a (300 MHz, $CDCl_3$):



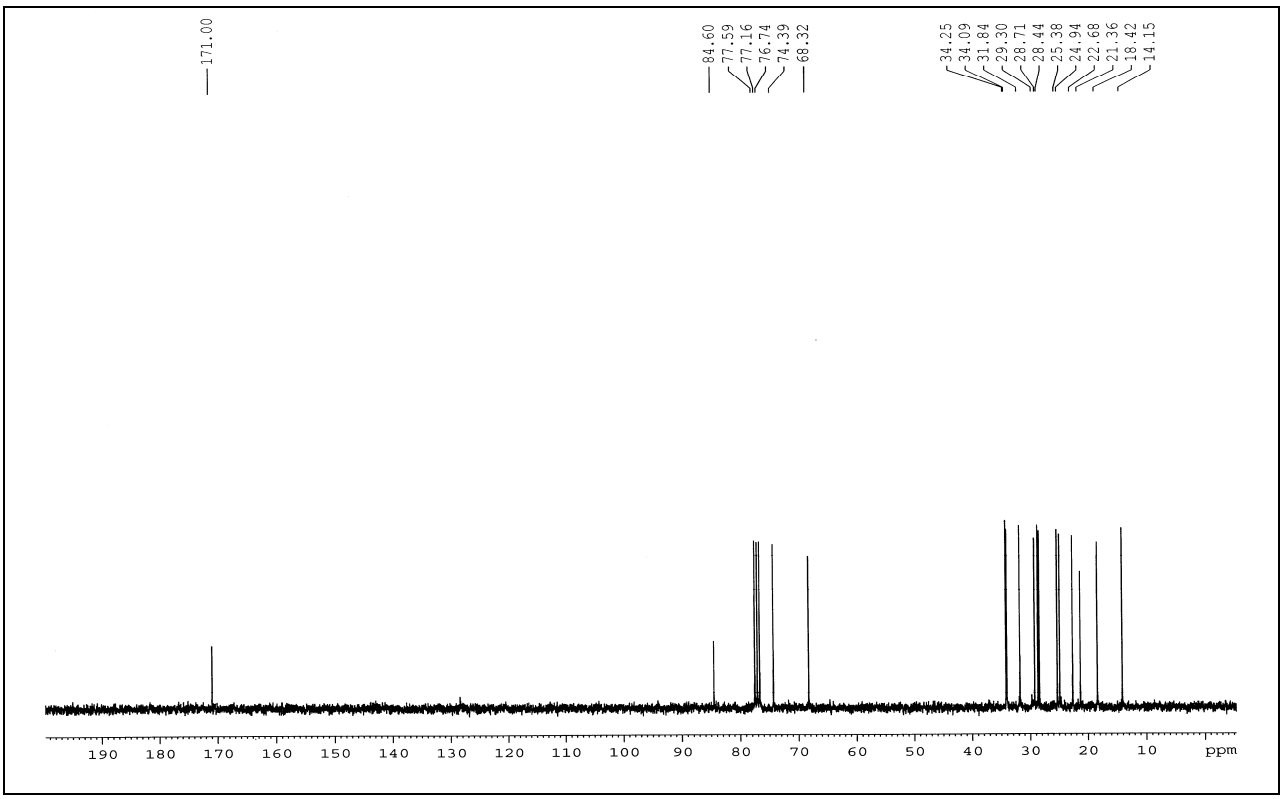
^{13}C NMR spectrum of 69a (75 MHz, $CDCl_3$):



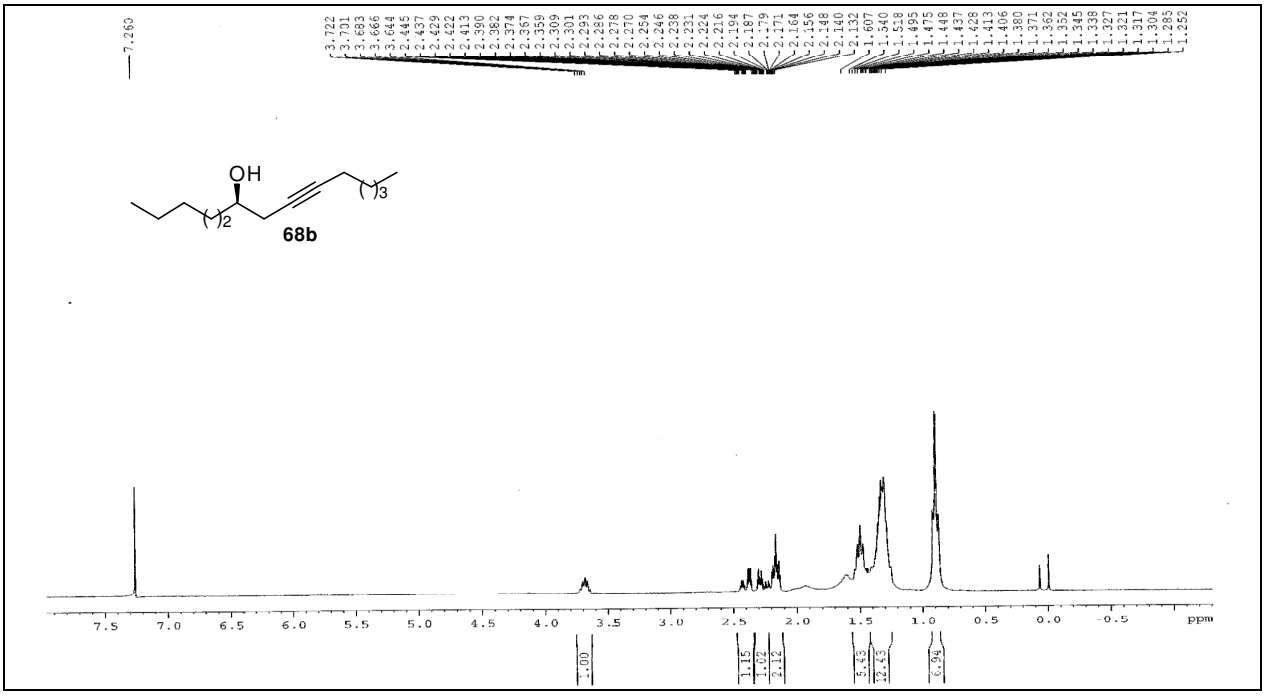
^{1}H NMR spectrum of 70a (300 MHz, $CDCl_3$):



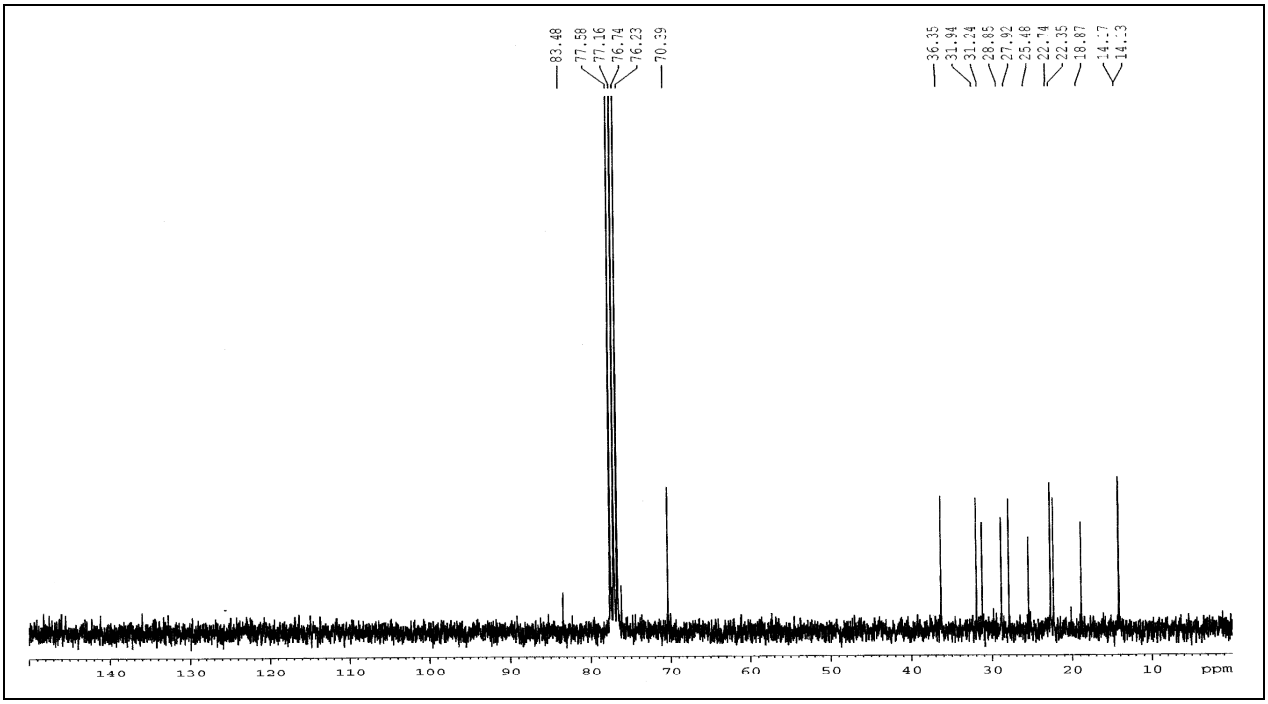
^{13}C NMR spectrum of 70a (75 MHz, $CDCl_3$):



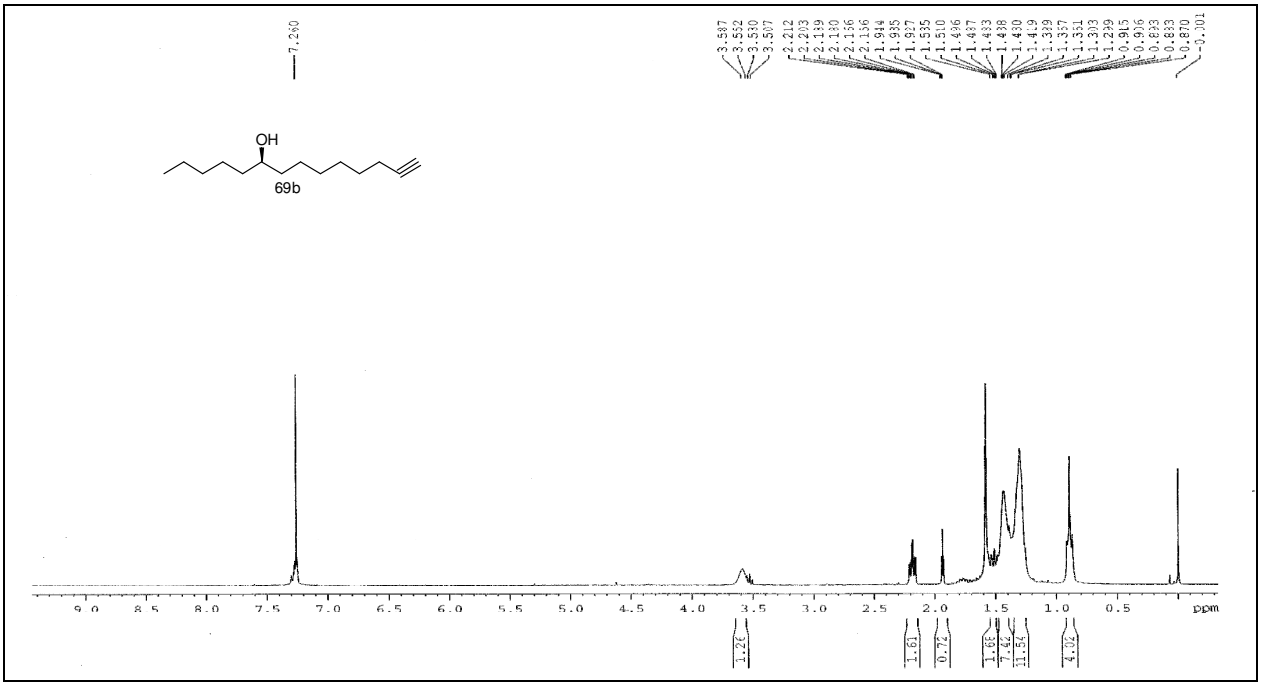
^{1}H NMR spectrum of 68b (300 MHz, $CDCl_3$):



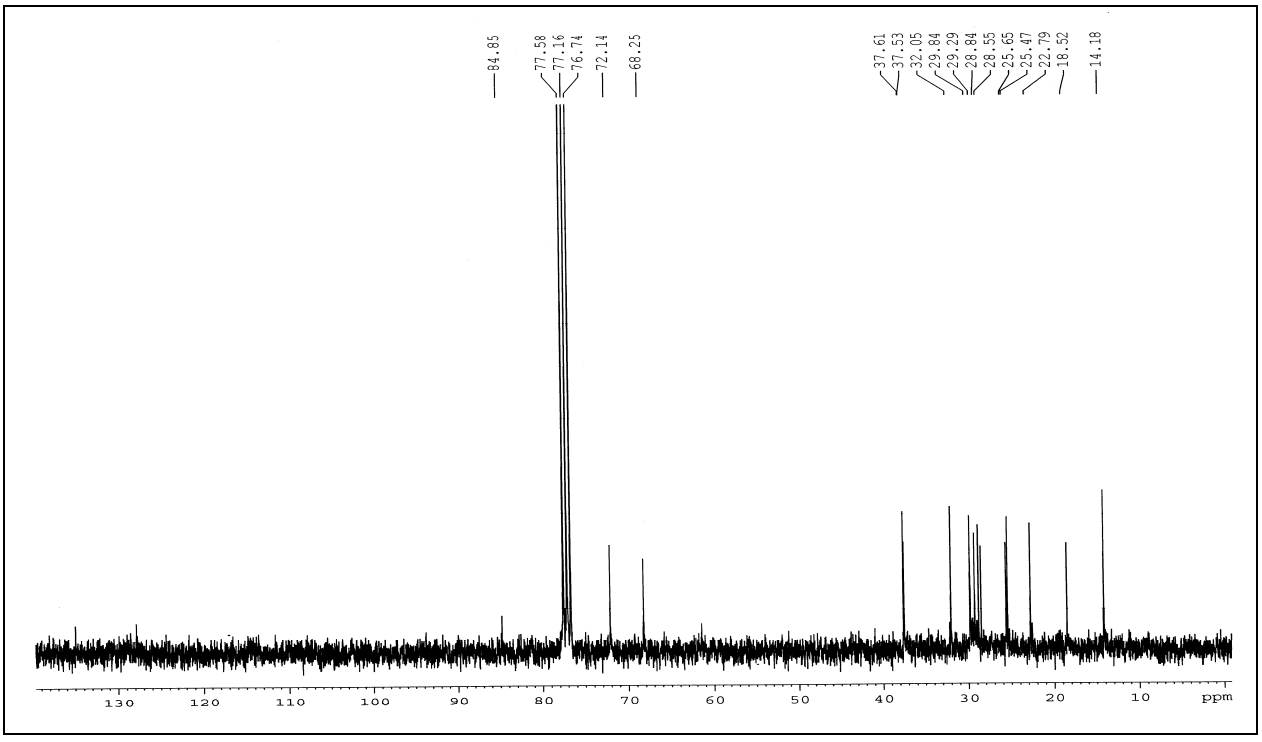
^{13}C NMR spectrum of 68b (75 MHz, $CDCl_3$):



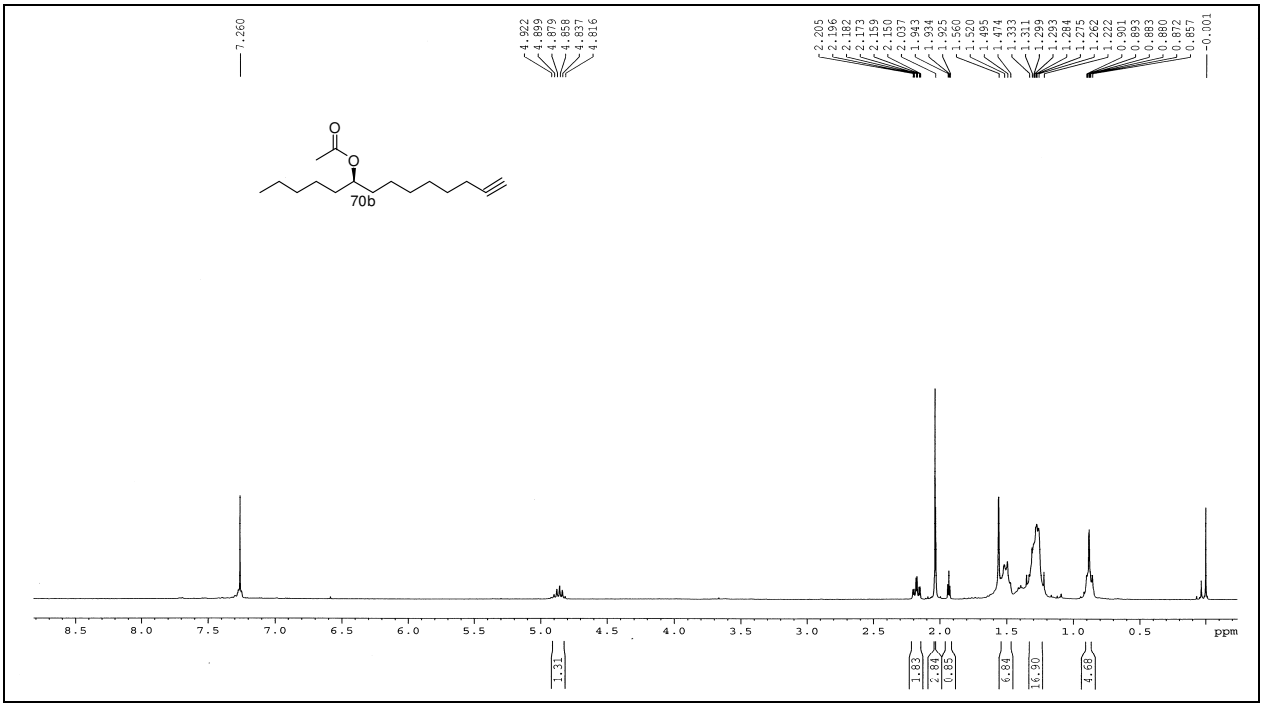
^{1}H NMR spectrum of 69b (300 MHz, $CDCl_3$):



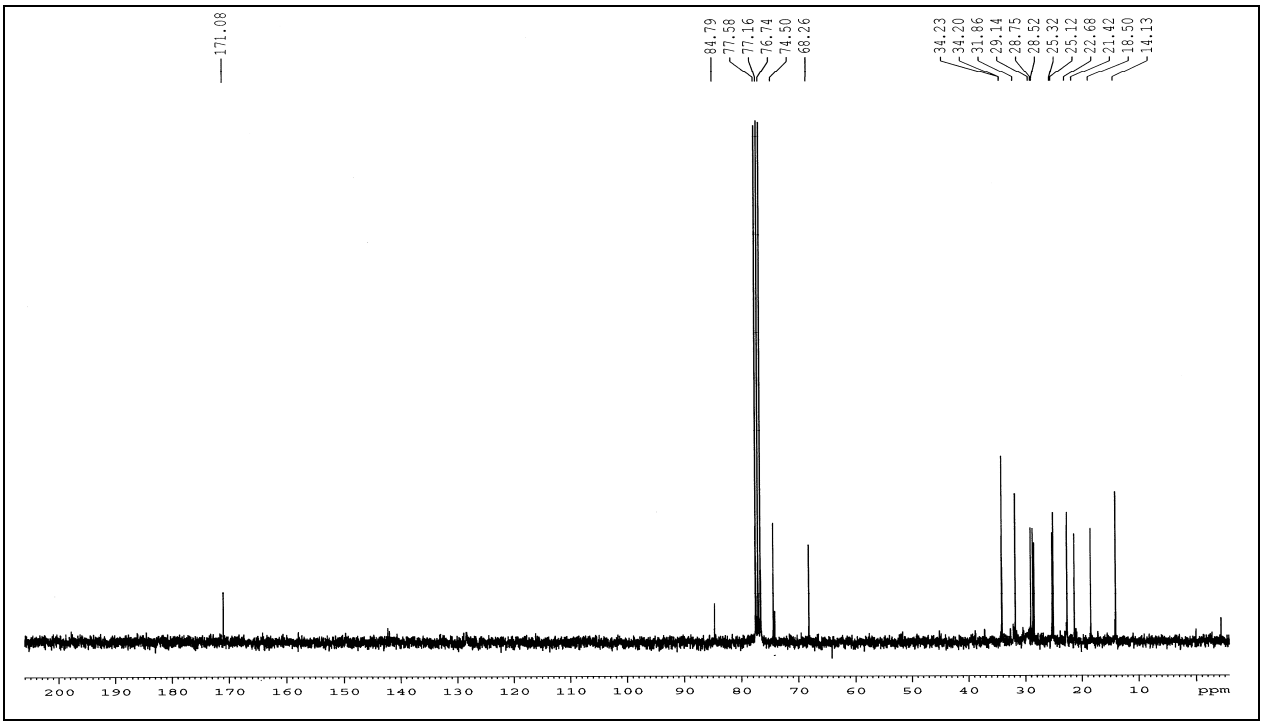
^{13}C NMR spectrum of 69b (75 MHz, $CDCl_3$):



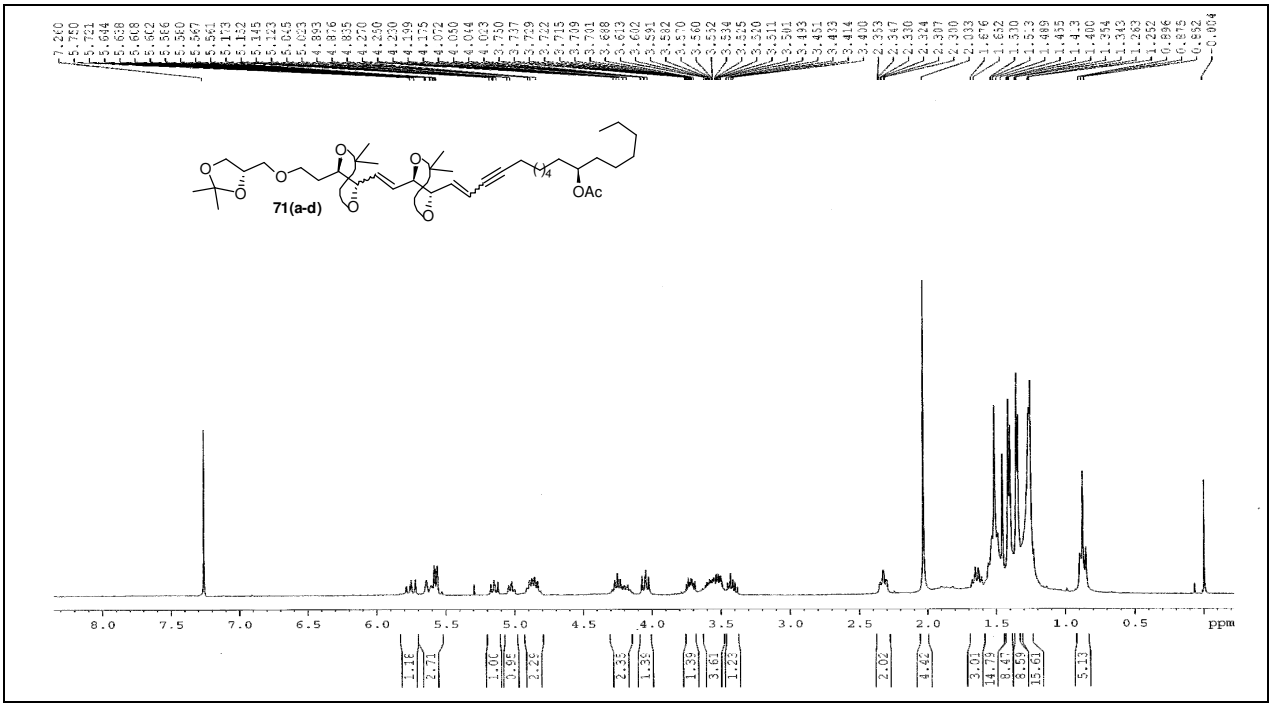
^{1}H NMR spectrum of 70b (300 MHz, $CDCl_3$):



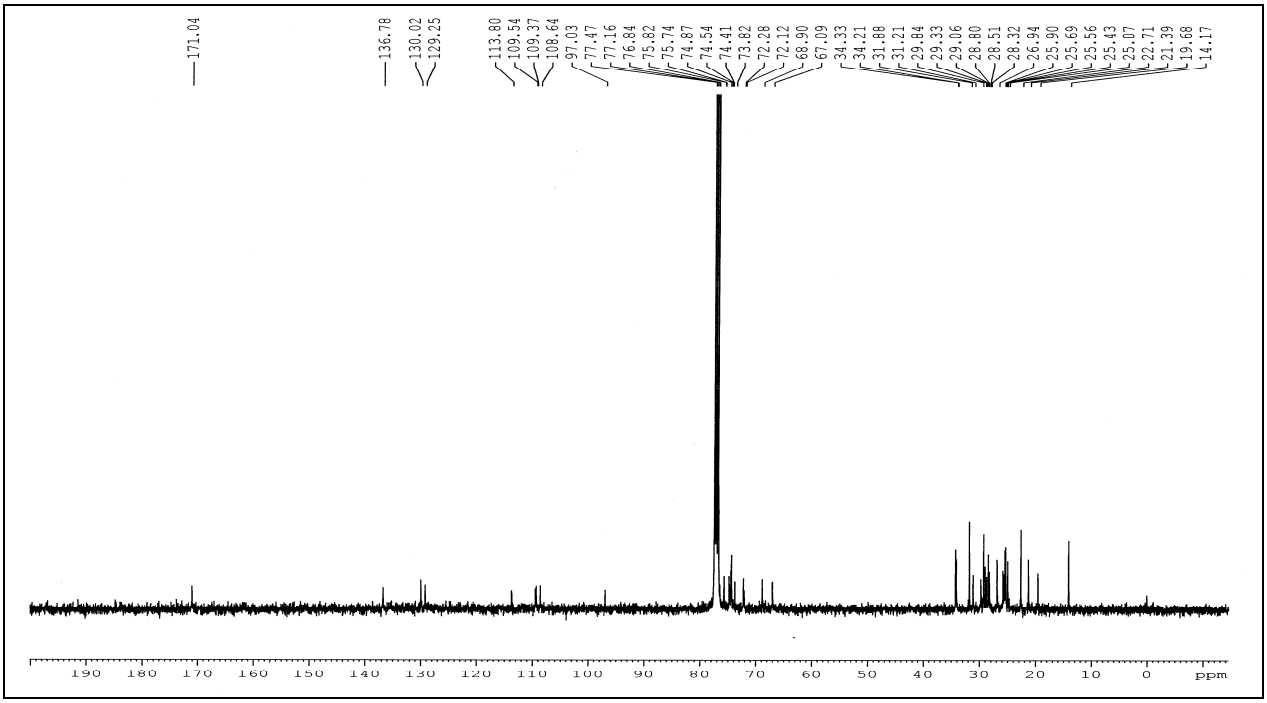
^{13}C NMR spectrum of 70b (75 MHz, $CDCl_3$):



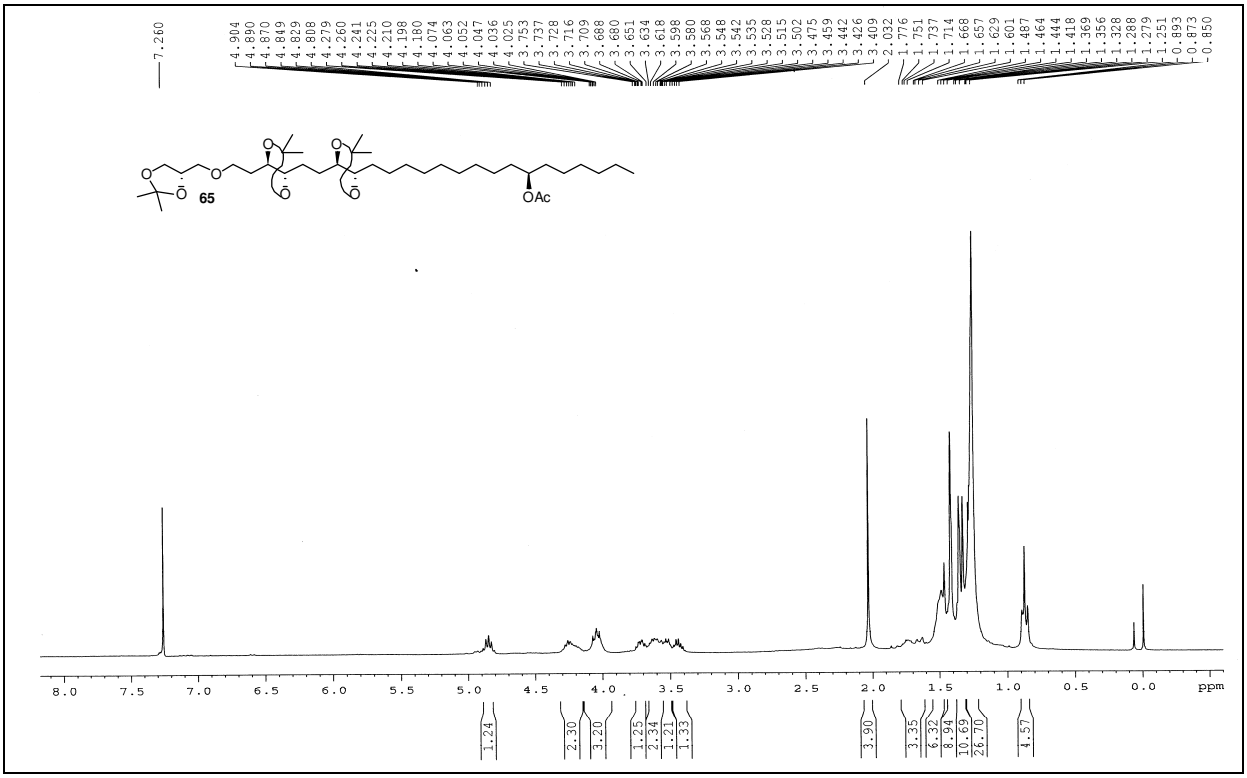
^{1}H NMR spectrum of 71 (a-d) (300 MHz, $CDCl_3$):



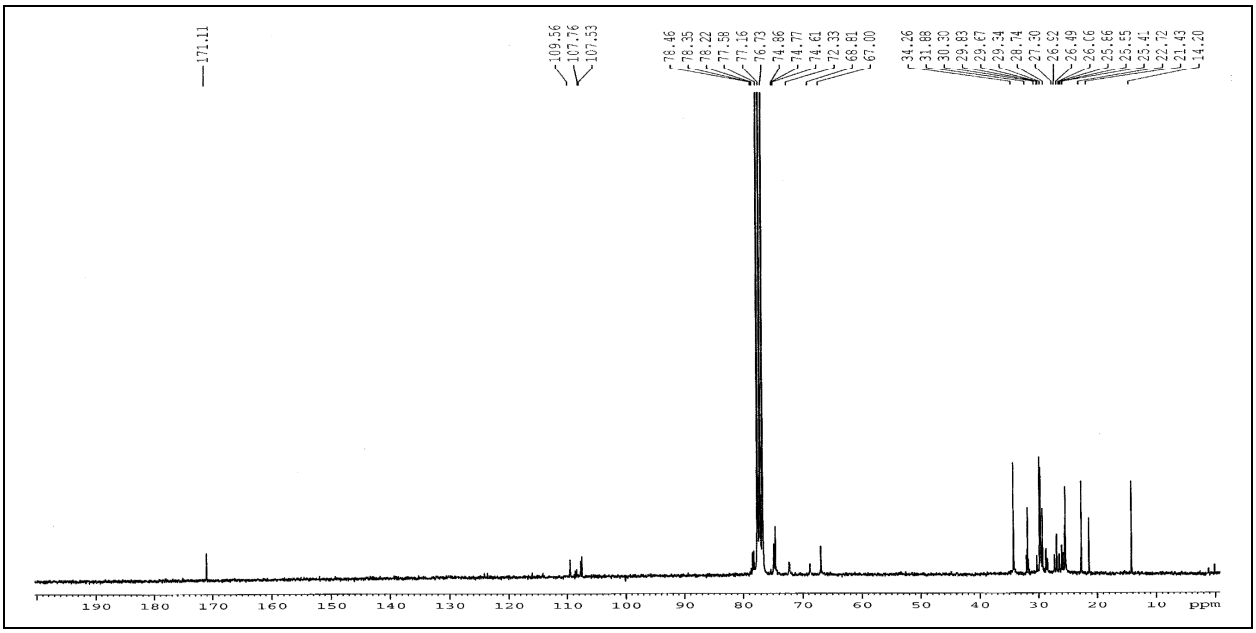
^{13}C NMR spectrum of 71 (a-d) (75 MHz, $CDCl_3$):



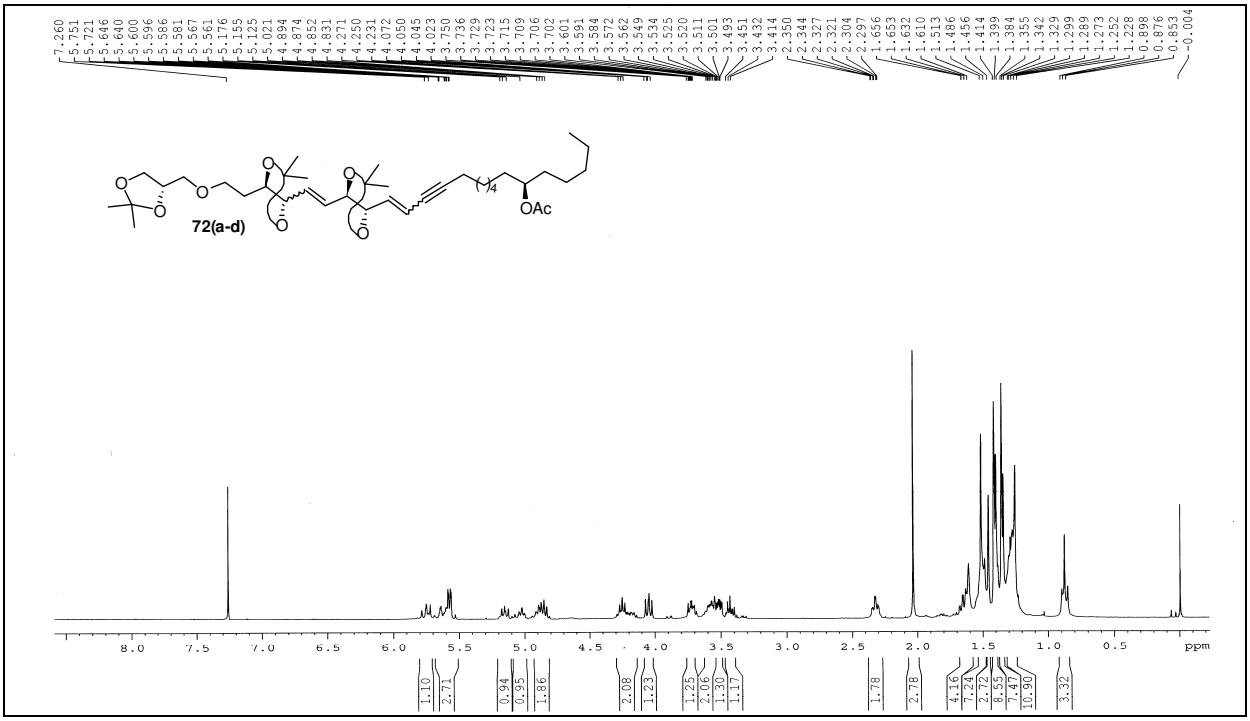
^{1}H NMR spectrum of 65 (300 MHz, $CDCl_3$):



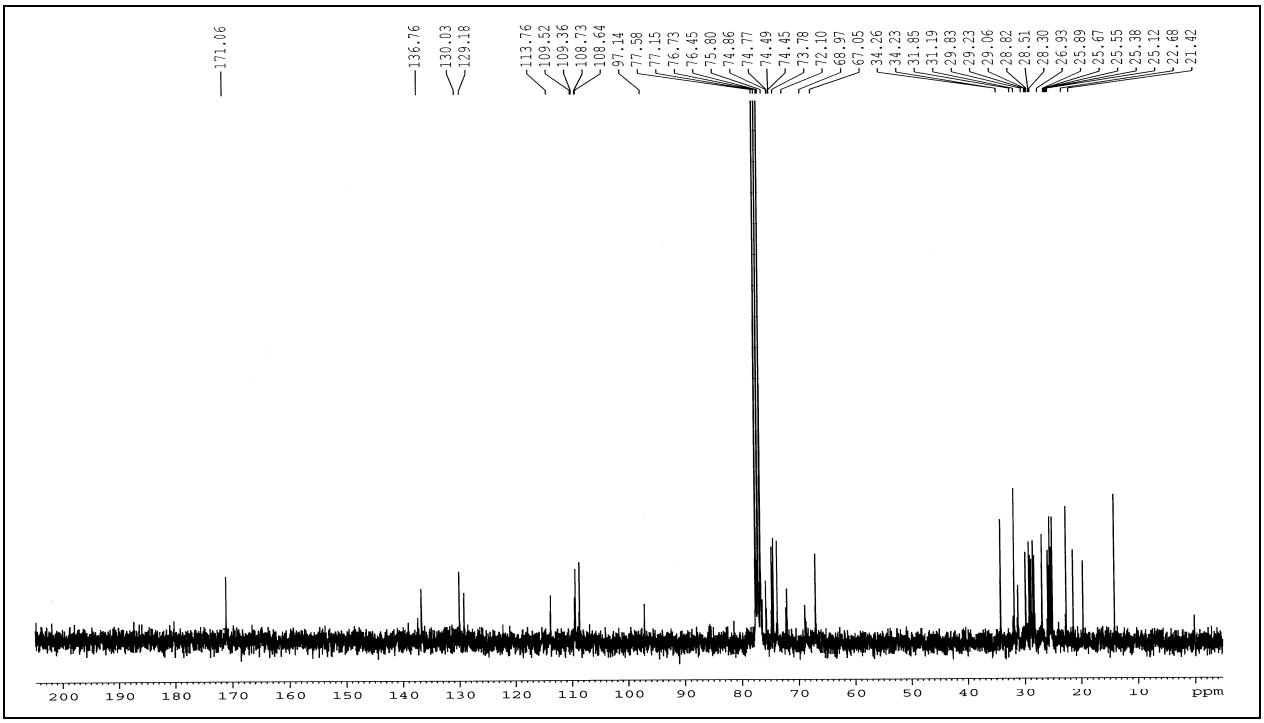
^{13}C NMR spectrum of 65 (75 MHz, $CDCl_3$):



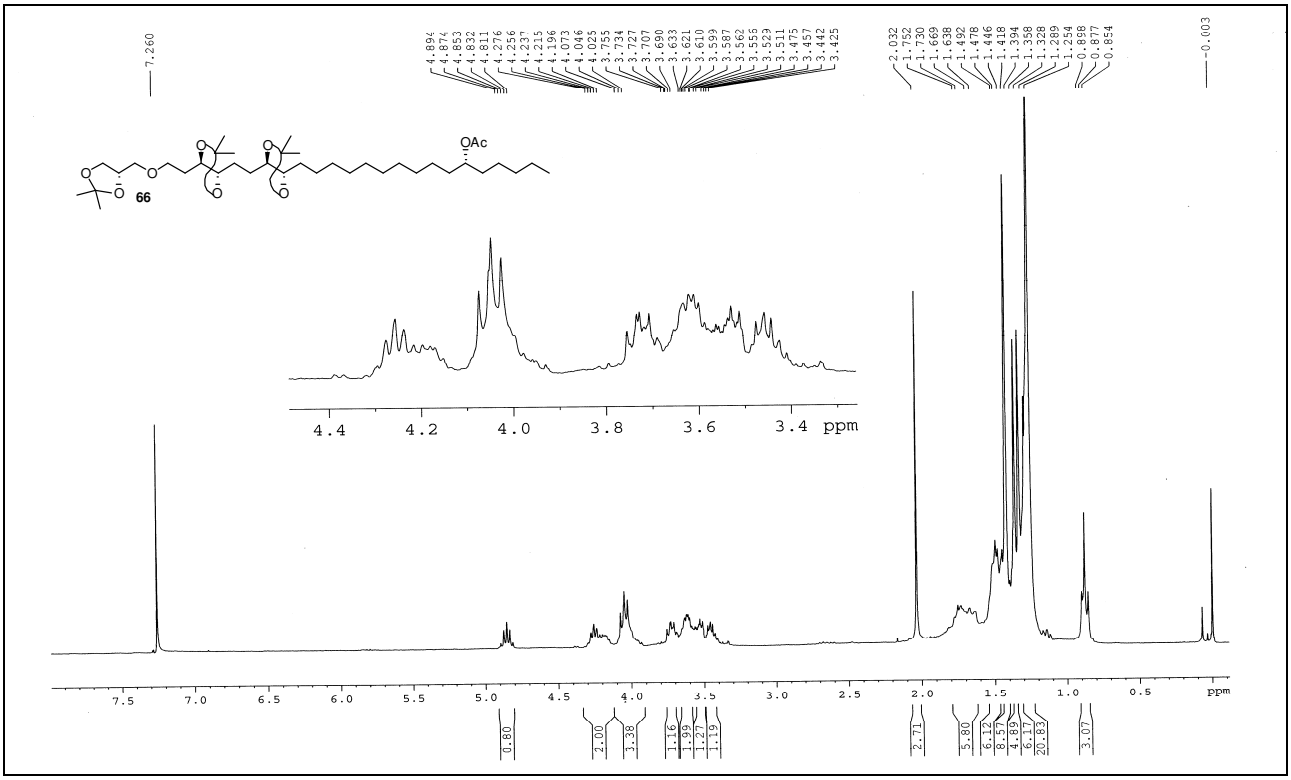
^{1}H NMR spectrum of 72 (a-d) (300 MHz, $CDCl_3$):



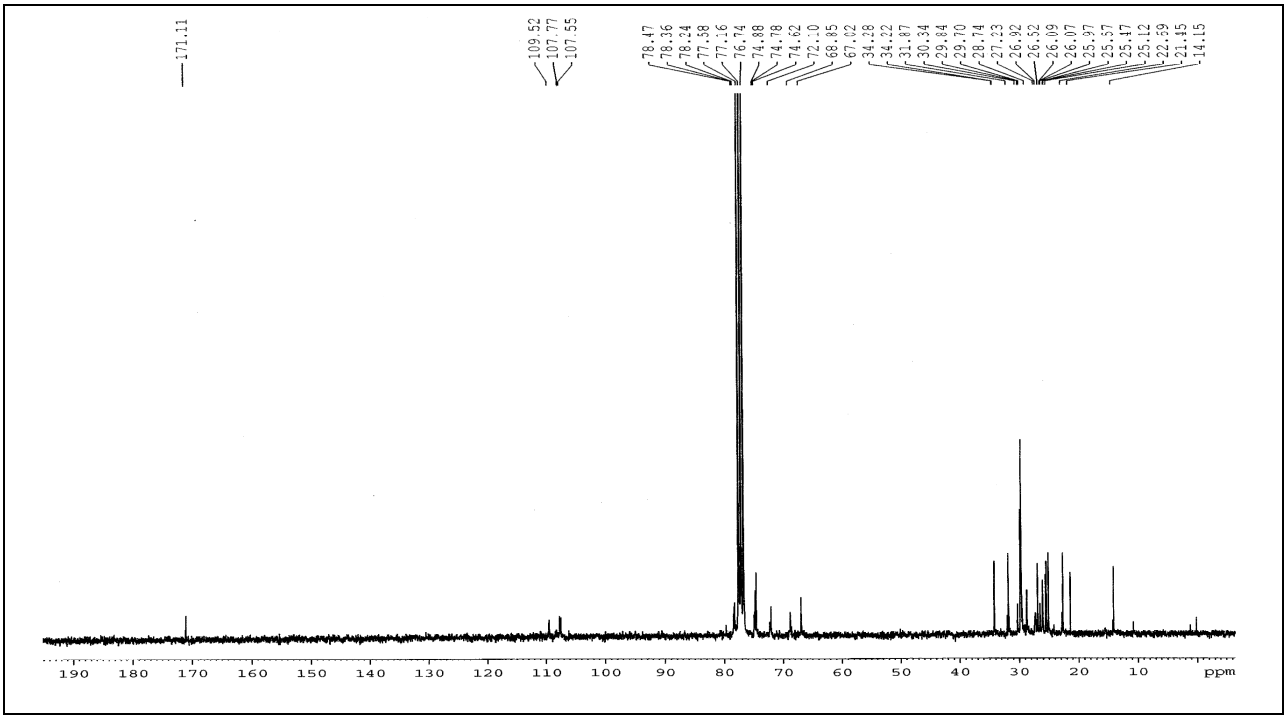
^{13}C NMR spectrum of 72 (a-d) (75 MHz, $CDCl_3$):



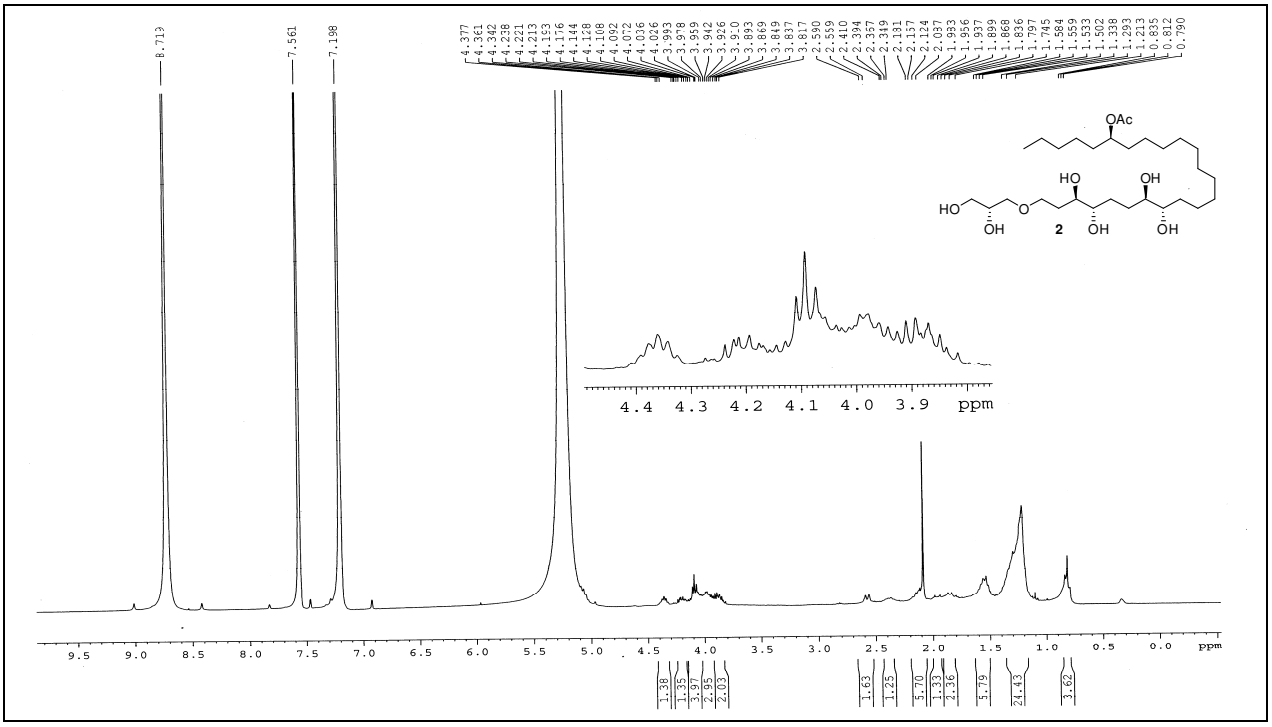
^{1}H NMR spectrum of 66 (300 MHz, $CDCl_3$):



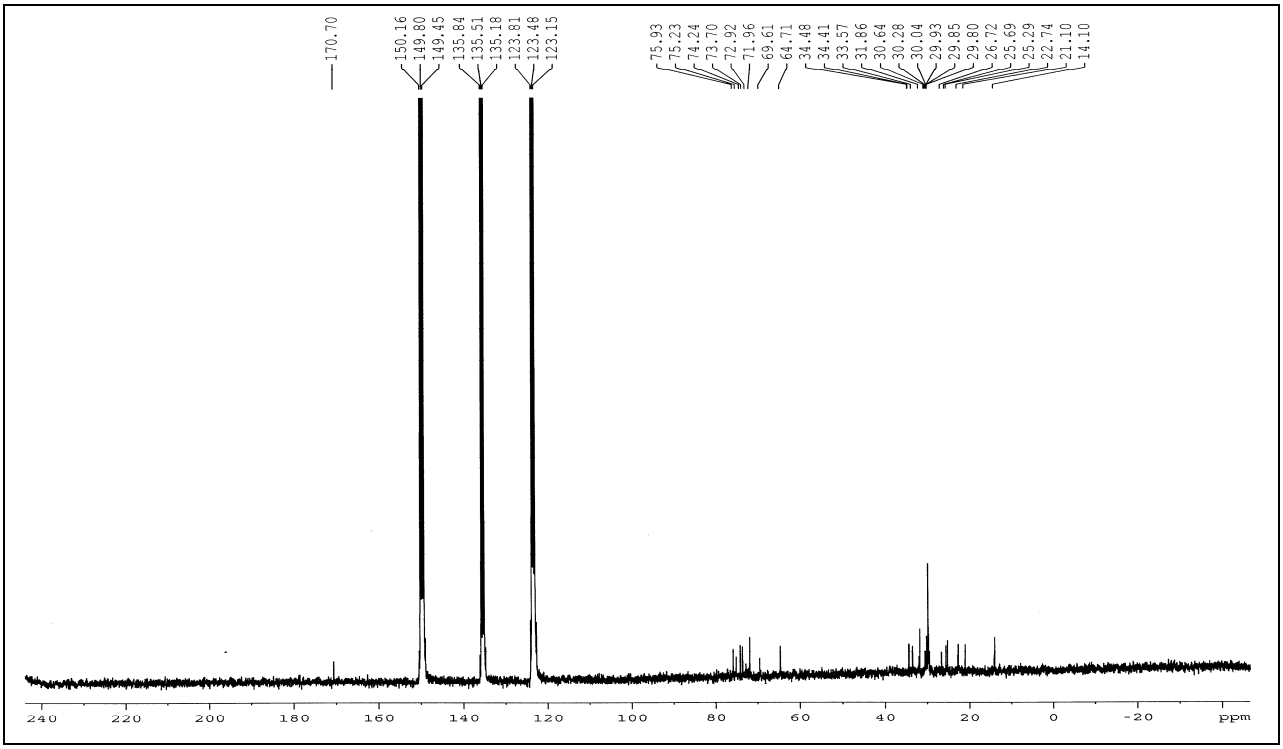
^{13}C NMR spectrum of 66 (75 MHz, $CDCl_3$):



^{1}H NMR spectrum of 2 (300 MHz, C_5D_5N):



^{13}C NMR spectrum of 2 (75 MHz, C_5D_5N):



HRMS spectrum of 2: